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ILLINOIS DEPARTMENT OF
PUBLIC HEALTH
A Healthier Today For A Better Tomorrow

John R. Lumpkin, M.D., Director

MEMORANDUM

TO: Craig Tarpoff, Chairman
Citizen Advisory Panel
Granite City, IL

FROM: Tom Long, Toxicology Program

DATE: October 15, 1991

RE: Soil Tilling Project
#411038801H

I have completed my review of the work plan provided for this project. In general, it appears to be a complete and well thought out approach to the problem. If carried out as stated, this project appears unlikely to pose any significant health concerns to area residents.

Some suggested clarifications and/or change include:

1) The work plan indicates property on which children live will be excluded. It was my understanding that this same stricture applied to property with neighboring children or to which children regularly visited (i.e., grandparents).

2) The type of security fencing seems inadequate. A higher, sturdier fencing would appear more appropriate.

3) The air sampling plan appears appropriate; however, I would suggest that background air sampling and meteorology be done for some time prior to beginning the sampling. This will serve to establish true background for the study and may have additional benefits later.

4) If it will not interfere with the study, I would suggest dust suppression steps be taken prior to and during tilling. This could involve a slight moistening of soil sufficient to hold down any dust generated.

5) The derivation of the action level for airborne dust is an interesting approach and I may steal it for future projects. I am unclear however, as to the rationale for the selection of the safety factor in equation 1 (Page 7, Appendix A). I can think of as many reasons for selecting 10 as 2 in the example provided and this needs clarification

or further discussion. Additionally, the background dust levels should be determined before locking into a derived level such as indicated. An alternate approach might be some increased percentage of background over a given time period will lead to shut down or more vigorous dust suppression steps.

Thank you for the opportunity to review this document and provide comment. Please call if there are any questions or further information is required.

lb/tlgcity3

REVISED FINAL DRAFT

STATEMENT OF WORK

SOIL HOMOGENIZATION PILOT STUDY

GRANITE CITY, ILLINOIS

Prepared For:

TECHNICAL COMMITTEE

NL INDUSTRIES/TARACORP SUPERFUND SITE

GRANITE CITY, ILLINOIS

NTH PROJECT NO. 13-1313-00

SEPTEMBER 24, 1991

GRANITE CITY

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MEMORANDUM

TO: Distribution

DATE: September 24, 1991

FROM: Sunil K. Agrawal Agrawal
Andrew B. Flynn
NTH Consultants, Ltd.

PROJECT NO.: 13-1313-00

SUBJECT: Statement of Work
Soil Homogenization Pilot Study
Granite City, Illinois

We have been instructed to provide the attached Statement of Work for the Soil Homogenization Pilot Study in Granite City, Illinois, for your information and review.

Subject to completion of the sampling activities, presently being conducted for the health study, it is anticipated that homogenization activities will commence during the week of October 7, 1991.

Therefore, any comments or suggestions that will enhance this study would be appreciated at your earliest convenience so they may be considered for incorporation into the Statement of Work prior to the commencement of homogenization activities. Your comments and suggestions should be directed to Mr. Steve Holt and Mr. Joseph Nassif at the addresses listed below:

Mr. Steve Holt
NL Industries, Inc.
Wyckoff Mills Road
P.O. Box 1090
Hightstown, NJ 08520
Phone: 609-443-2405
Fax: 609-443-2374

Mr. Joseph Nassif
Coburn, Croft & Putzell
One Mercantile Center
Suite 2900
St. Louis, MO 63101
Phone: 314-621-8575
Fax: 314-621-2989

Thank you for your cooperation.

Distribution:

The Honorable Von Dee Cruse, Granite City
Ms. Susan Bacon, Granite City
Mr. Brett L. Hanke, Granite City
Mr. Craig Tarpoff, Granite city CAC
Dr. Thomas Long, IDPH
Dr. Renate Kimbrough, IEHR
Ms. Kathleen Andria, H.E.L.P.
Mr. George Webb, Taracorp

cc: Granite City Steering Committee

ILLINOIS DEPARTMENT OF
PUBLIC HEALTH
A Healthier Today For A Better Tomorrow

John R. Lumpkin, M.D., Director

August 2, 1991

Kathy Andria
1729 Maple St.
Granite City, IL 62040

Dear Ms. Andria:

Pursuant to my conversation with you and Craig Tarpoff, I am setting down some of my concerns over the reported plan to allow experimental soil treatment to be conducted in some local parks and playgrounds as a measure of its potential efficacy in remediating lead contamination. One of my chief concerns is, of course, for the health study that we have all labored so long to develop and complete. It is vitally important for the purposes of this study that exposure to lead in the various environmental pathways be as close to normal as possible if we are to draw any meaningful conclusions from the information collected and keep potential criticism of the study to a minimum. Tilling, digging, or otherwise disturbing the soil raises the possibility of altering the exposure patterns for some (perhaps many) individuals. While I support the concept behind the proposed experiment, I do not think that it has to be done now or even necessarily in the area. An additional study-related concern is simply the number of competing activities planned for the area by various organizations and agencies due to the environmental problems associated with this site. Our study depends on the voluntary cooperation of the citizens of Granite city, Madison, and Venice, but I see an increasing risk of confusion over who is doing what, where, and when. With uncertainty comes a reluctance to cooperate and once again this puts our study at risk. I believe only one activity ought to be carried out at a time to reduce this possible source of confusion. In the interests of making our study as strong as possible to the benefit of all concerned, I believe that this experiment should at least be delayed until after we have completed our efforts.

Another concern is over the selection of the proposed test areas. As you are aware, our main concern with lead contamination is for its deletrious effects on children. I cannot think of a more likely place to find children than in a park or playground. The proposed plan raises the possibility that children may be exposed to lead during or after the experiment. The disturbance of soil, raising of dust, and the natural curiosity of children around the attractive nuisance of a construction site make increased exposure to lead a concern. Additionally, the nature of the test suggests the possibility that physical hazards might be created. I am not sure who would be liable for any actual or perceived injuries or damages that might result from these experiments, but I believe that this is something that the park board and/or city may wish to consider before granting final approval. Whatever the merit of this proposal, the wisdom of using property in which people, particularly children, congregate seems questionable. In this light, the use of other property in Granite City or in another area would seem advisable.

In any such situation, the organization conducting the effort develops a work plan which defines the scope of action, details the work to be performed, how, where,and when it is to be done, who is to do it, what steps are being taken to reduce or eliminate risks, contingency plans for accidents or unforeseen events, the rationale behind the proposal , and the like. It would seem to be in the best interests of the park board and/or city to have such a document to be developed and reviewed before the experiment is permitted to procede. I don't think its an unreasonable request to those proposing the study to provide such a work plan to the city. I would also be happy to review such a plan on behalf of the city and provide comments or other such input as might be necessary to protect all involved. In light of the fact that USEPA's plans do not call for work to begin before 1993, I believe that there is more than sufficient time to prepare such a document, review it, and still carry out the work if that is the desire of the city. Such steps can only serve to improve the study while protecting the residents from possible hazards and the city from liability.

In summary, I believe this proposal has merit, but that it should be delayed until after the health study is complete and that the use of parks and playgrounds ought to be re-considered. As there is no real reason to conduct such tests within the city limits, the proponents of the experiment may wish to re-consider the location(s) for the experiment entirely.

I hope this letter is helpful in clarifying my comments. Please feel free to contact me if questions remain or further information is required.

Sincerely,

Thomas F. Long
Senior Toxicologist
Environmental Toxicology Program

cc: Craig Tarpoff, Chairman
Citizen's Advisory Panel

Von Dee Cruse, Mayor
Granite City

Division of Environmental Health
Region 4, Edwardsville

Renate Kimbrough, M.D.
Institute for Evaluating Health Risks

Central Office File

1.0 INTRODUCTION

NTH Consultants, Ltd. (NTH) has been retained by a group of industrial clients to conduct a pilot study to evaluate the effectiveness of homogenizing soil to redistribute lead in near-surface soils (0"-9"). This pilot study will be used to evaluate the applicability of this technology to selected residential soils. Addition of additives such as lime and nutrients (fertilizers) will also be evaluated for effectiveness in reducing the mobility of lead in the near-surface soils and for the promotion of vegetative growth. A laboratory bench top study will be used to optimize the dosage of additives prior to implementation in the pilot study.

1.1 Site Description

The pilot study will incorporate selected properties located within Madison and Granite City, Madison County, Illinois, containing lead in the surface soils (0"-3"). The surface soils of the selected properties may have been impacted by airborne particulates containing lead as a result of various lead smelting activities in the past at the NL Industries/Taracorp site, and by other sources of lead including motor vehicle fuel emissions.

1.2 Background

The test plots to be selected for the Soil Homogenization Pilot Study are within or adjacent to the area classified by the United States Environmental Protection Agency (EPA) as a Superfund Site and is known as the NL Industries/Taracorp Superfund Site. The exact extent of lead concentrations in residential surface soils has not been determined at this time.

2.0 OBJECTIVES

The primary objectives of this pilot study are (1) to determine the optimum method for obtaining homogeneous distribution of lead throughout the test plots, (2) to measure the effect of additives, such as lime on the mobility of lead and nutrients on the vegetative growth, and (3) to optimize environmental controls that will be used to protect the local population and environment. Homogenization will be performed utilizing tilling/ mixing equipment in selected plots that are representative of typical lead distributions. To achieve these objectives, the following tasks will be performed:

1. Develop a relationship between the number of passes (using tilling/mixing equipment) and homogeneity of total lead concentrations in the soil. The soil will be considered relatively homogeneous when the mean and standard deviations of lead concentrations from consecutive passes approach constants.

2. Establish criteria for applicability of the homogenization process to the residential soils in the Granite City area.

3. Evaluate the effectiveness of additives such as lime and nutrients in reducing the mobility of lead from the soil and to enhance vegetative growth.

4. Determine the optimum methods for applying nutrients and lime to the near-surface soils.

5. Monitor air quality throughout the pilot study to optimize environmental controls and to determine the actual emission factors associated with the homogenizing operation. These data will be used to evaluate the potential impact on public health of the homogenizing process, as compared to an excavation and backfilling process.

6. Evaluate the significance of the data and determine if the pilot study can be adapted to a full scale application of this technology.

3.0 SELECTION OF PLOTS

Test plots within the project area will be used to perform the field homogenization study. A number of potential test plots will be identified. All potential test plot soils will be sampled and analyzed by X-ray Fluorescence (XRF) methods for total lead concentration in accordance with the procedures in Section 4.0. Plots for use in the study will be selected based on availability, soil characteristics and distribution of lead in near-surface soils. Properties where young children live will not be used for this study. The methods that will be used to evaluate and select the test plots are described in the following sections.

3.1 Screening of Potential Test Plots

Potential test plots will be screened using XRF methods to establish relative soil lead concentrations. Soil lead concentrations will be estimated with the XRF instrument using a pre-established calibration curve. The screening will provide preliminary information and data for the selection of the test plots. Split soil samples will be obtained and sent to the analytical lab for total lead analysis (EPA Method 7420) during the screening process for calibration of the XRF and QA/QC purposes. Sampling procedures and XRF analysis are discussed in detail in Section 4.

3.2 Selection Criteria

The 16 plots will be selected based on the following criteria:

- Plots must be located in areas which represent typical residential and industrial properties that have been impacted by various lead smelting activities at the NL Industries/Taracorp site.

- The mean soil lead concentration in the surface soil samples must range between 500 and 2,000 mg/kg.

- Soil lead concentrations must decrease with depth. The mean of all the samples, 0-3", 3"-6" and 6"- 9" sample intervals, must be less than the 0-3" concentration. Commonly, lead originating from a smelter and deposited on the ground will be highest in the surface soil horizon (0"-3") and decrease with depth.

- Properties where young children live will not be used for this study.

Preferably, the selected test plots will also exhibit a range of soil lead variability (slightly to highly variable) as measured by the standard deviations of the measured concentrations. The effect of this factor on tilling/mixing efficiency may also be evaluated. In the event that plot selection is limited by availability, the selection criteria and evaluation techniques may be modified.

3.3 Number of Test Plots

Ideally, the number of test plots needed for this project is based on the number of variables that will be evaluated. The following variables have been considered for this pilot study:

- Homogenization method - Two types of tilling/mixing equipment will be used during the study and the effectiveness of each type will be evaluated.

- Soil Lead Concentration - Test plots will have initial mean surface soil lead concentrations within two ranges, either between 500 and 1,000 mg/kg or 1,000 mg/kg and 2,000 mg/kg.

- Lime and/or nutrient addition - Lime addition is one of the variables to be studied. Lime will be added to half of the selected test plots. Nutrients will be added to all of the selected test plots, therefore nutrient addition does not represent a variable.

In order to represent all possible combinations of the three variables, a minimum of eight (8) test plots will be required. To evaluate reproducibility of the results 8 additional test plots with similar combinations of variables will be selected, making the number of required test plots sixteen (16). Figure 1, Plot Selection Criteria, shows a schematic diagram of the 16 test plots and their associated variables. This design may be modified based on characteristics of available potential test plots.

4.0 SOIL SAMPLING AND FIELD ANALYSIS

Soil sampling will be conducted throughout the project to determine the effectiveness of soil homogenization and evaluate the changes in soil lead concentration and mobility over time. Sampling will be performed prior to and after soil homogenization, and again approximately one year later. Details of the soil sampling methodologies and field X-ray Fluorescence (XRF) analysis procedures for total lead are presented below.

4.1 Sampling Events

Four sampling events will occur during the pilot study: (1) screening plots for selection, (2) immediately prior to the homogenization operation, (3) immediately after soil homogenization, and (4) nine months/one year after the soil is homogenized. Additionally, samples will be taken periodically during the homogenization operation for quality control.

4.1.1 Screening of Potential Test Plots

Potential test plots will be screened using XRF methods to estimate soil lead concentrations. A grid will be established for each potential test plot to ensure that sample points can be located and referenced if the test plot is selected. A typical sampling grid is shown in Figure 2, Proposed Sampling Grid. The grid spacing and geometry may change based on the dimensions of the selected test plot. At least one temporary benchmark will be utilized for establishing sample grid locations and relative elevations.

Eight (8) soil cores will be obtained at the grid locations in each proposed test plot using a nine (9) inch sampling probe. Each soil core will be separated into three (3) samples as shown in Figure 3, Soil Core/Label Example. The soil samples will represent depth intervals of 0-3", 3"-6", and 6"-9". During the screening process, these soil samples will be analyzed by XRF to determine the approximate soil lead concentrations. The screening will provide background information and preliminary data for the selection of the test plots.

4.1.2 Initial Sampling of Test Plots

Eight soil cores will be obtained from each test plot using the nine inch sampling probe, as per section 4.3. The soil cores will be separated into 3 samples, as discussed above, and submitted for XRF and laboratory analysis for total lead (EPA Method 7420) immediately prior to initiating the soil homogenization. This sampling event will provide baseline information to evaluate the results achieved after homogenization.

A total of five (5) composite soil samples will also be obtained during the initial sampling to evaluate the mobility of lead and to support the Bench Top Study required to identify the optimum dosage of lime and nutrients, see section 5.0. Lead mobility will be evaluated using the Toxicity Characteristic Leaching Procedure (TCLP), EPA Method 1311. Each composite soil sample will be prepared by obtaining soil cores at 8 separate locations in a test plot. Five test plots will be selected for the composite samples.

4.1.3 Sampling Between Passes

Six soil cores may be obtained from the test plot after each pass performed with the tilling/mixing equipment. The soil cores will be separated into two samples that represent depths of 0"-3" and 3"-9". These samples will be analyzed using the XRF. Selected soil samples will be submitted for analytical testing to verify the performance of the XRF and to support instrument calibration as shown in Table 4 of Appendix B. Samples obtained between passes will be used to determine the optimal number of passes required to homogenize the soil.

4.1.4 Post-Homogenization Sampling

After the homogenization of the test plots has been completed, as discussed in Section 6.0, the soils in the test plots will be re-sampled. The post-homogenization sampling program will begin by locating the sampling grids established during the initial soil sampling program, as discussed in section 4.2. This will be accomplished by utilizing the temporary benchmark placed prior to the initial sampling event. Field personnel will also utilize field drawings and photographic records in order to confirm sampling grid locations.

Once the sample locations are determined, soil cores will be obtained. The soil cores will be separated into discrete soil samples representing depths of 0"-3", 3"-6" and 6"-9". The discrete samples will be chemically analyzed for total lead concentration.

Five composite soil samples will be obtained from the test plots originally selected for composite samples. The composite samples will be chemically analyzed to evaluate the mobility of lead (TCLP) in the homogenized soil.

After the composite soil samples are obtained, lime will be applied to half of the plots in the pilot study. All of the test plots will have nutrients applied to enhance vegetative growth.

4.1.5 Post Homogenization Soil Sampling; After 9 Months/1 Year

Approximately one year after the post homogenization soil sampling portion of the pilot study has been completed, the test plots will be re-sampled and analyzed to determine total lead concentration and lead mobility (TCLP).

Eight soil cores will be obtained from each test plot and separated into discrete samples from depth intervals of 0"-3", 3"-6" and 6"-9". The discrete soil samples will be tested for total lead concentrations at an analytical laboratory.

Five composite soil samples will also be obtained at the original five selected test plots to evaluate the mobility of lead (TCLP) in the treated soil. Eight soil cores from each test plot will be used to make one composite sample.

4.2 Location of Samples

During the selection of test plots a sampling grid will be established to reference sample locations. After test plots have been selected for the pilot study, permanent grid location points will be set. Grid location points will be set using at least one concrete nail or local structure as a temporary benchmark.

Additional nails will be set if the conditions at the test plot site are favorable.

Soil cores will be obtained from each test plot at locations based on a simple grid pattern as shown in Figure 2, Proposed Sampling Grid. Using systematic grid sampling, a minimum of eight sampling points will be located within each individual test plot horizontally. The grid pattern and grid spacing may vary depending on the size of the test plot. Additional sampling locations may also be needed to ensure validity of the statistical evaluation (as discussed in Section 8.0).

Each sampling event will use the same grid points to re-establish core locations. Prior to the initial sampling event, field drawings and photographs of each test plot will be recorded by field personnel thereby documenting the associated grid pattern and core locations. These documents will assist in re-establishing identical core locations for each subsequent sampling event.

4.3 Sampling Procedures

Soil sampling of the selected plots will be performed by the following method. Generally, soil cores will be obtained by using a stainless steel sampling probe equipped with plastic liners. The sampler will be advanced into the soil to a depth of 9 inches.

Upon retrieval of the soil core, the liner containing the soil will be removed from the sampling probe, capped and labeled, as shown in Figure 3, Soil Core/Label Example. Discrete soil samples will be obtained for each depth interval (0"-3", 3"-6" and 6"-9") by separating the soil core at the appropriate location. This will provide 24 discrete soil samples per test plot.

Five (5) composite soil samples will be collected for the bench top study. One composite soil sample will be obtained from each of the 5 selected test plots using the stainless steel sampling probe described above. The sampling probe will be advanced to a depth of 9 inches. The soil core obtained will then be placed in an eight (8) ounce jar and labeled. The probe will be used at the same location until the eight ounce jar is filled. Soil cores will be obtained at the 8 pre-established grid locations and mixed at the laboratory to develop one representative composite sample from each of the five selected test plots.

The sampling probe will be decontaminated between each sample core by washing with an Alconox and de-ionized water solution followed by a final rinse with de-ionized water.

4.3.1 Sample Data Sheets

Soil sampling information will be recorded directly onto preformatted sample data collection forms. The forms will be completed in the field by the individual assigned to the specific sampling task. An example of the Sample Data Sheet is provided in Figure 4.

The Sample Data Sheets will be collected and validated by the field supervisor or by another field staff member if the field supervisor performs the sampling. Information from the sampling forms will be subsequently transferred into the data management system discussed in Section 7. Validated Sample Data Sheets will be sent to the NTH Farmington Hills office at the end of each work week where they will be maintained in a binder in chronological order.

4.3.2 Sample Designation and Documentation

Each plastic soil core container will be labeled in the field with the project number and a sample identification number. The sample identification number defines the area (plot) sampled, sample location, sample depth, date sampled and sampler identification. An example of the sample identification number is shown below:

01-001A-071891-150

Where:

Sample plot: 01

Sample location: 001

Sample Depth: A (0-3")

Date sampled: 071891

Personnel ID: 150

The sample label will indicate the top of the soil core with an arrow, enabling the required sample depth intervals, 0-3", 3"-6" and 6"-9" to be labeled as "A", "B" and "C" respectively. The sample label is shown in Figure 3.

The collection of each soil sample will be documented in the field with a photograph, sample data sheet and chain-of-custody record. Chain-of-custody for the soil samples will be conducted in accordance with the procedures outlined in the following section. If a sample is analyzed by a subcontracted analytical laboratory, sample custody will become the responsibility of that analytical laboratory upon receipt.

4.3.3 Chain-of-Custody Procedures

To maintain and document sample possession, chain-of-custody procedures are followed. The field sampler will be personally responsible for the care and custody of the samples collected until they are properly transferred or dispatched to the analytical laboratory.

Prior to the submittal of the samples to the analytical laboratory, sample custody will be the responsibility of NTH and will be controlled by the use of chain-of-custody procedures. At the completion of each sampling day, the field crew will relinquish custody of the samples and all documentation forms to the on-site field supervisor. The custody transfer will be documented by a record of the date, time, and signatures on the chain-of-custody form. The chain-of-custody form will also include the date of shipment, sample identification number and a list of requested analytical tests. This form functions as an official communication to the laboratory and identifies the particular analyses required for each sample, and provides further evidence that the chain of custody is complete.

A copy of the chain-of-custody will be retained by NTH, and the original chain-of-custody form will be enclosed in the transportation container with each sample shipment. An example of a standard NTH chain-of-custody form is presented as Figure 5, Chain-of-Custody Record Form.

4.3.4 Sample Shipping Procedures

A sample tracking log will be completed at the end of each day. An example of this log is presented as Figure 6, Sample Tracking Log. The form will be used to track the sample through collection, storage, and shipment to the laboratory. The sample shipping procedures are described in the following paragraphs.

Samples that are kept on-site will be stored temporarily prior to shipping in compliance with standard EPA protocol. A designated locking storage closet will be used for this purpose. When samples are to be shipped to the analytical laboratory for analysis, the delivery service will be notified for pick-up. The field supervisor will release the custody of the samples to the delivery service personnel. The transfer will be documented by a signed receipt and chain-of-custody form at the time of transfer. The delivery service will assume an insured responsibility for the custody of the samples until delivery at the analytical laboratory has been completed. The laboratory will then sign a receipt for the delivery, and the chain-of-custody form which will document the transfer of the sample custody to the analytical laboratory.

Copies of the sample tracking logs, chain-of-custody forms and laboratory assignment request forms will be collected at the end of each day by the field supervisor. The laboratory assignment request form is shown as Figure 7. Each bound package of forms will represent all samples shipped on that particular day. These records will be sent to the NTH Farmington Hills office at the end of every week. Each days' shipment will then be assigned a shipment number and filed sequentially in the shipment logbook.

4.3.5 Sample Shipment and Consignment

Sample shipments from the site to the analytical laboratory will be made in compliance with Department of Transportation (DOT) CFR 45 regulations. All samples for chemical analysis will be shipped in protected coolers by the delivery service to Martel Laboratory Services, Inc. in Baltimore, Maryland. Each shipment will contain the original "Chain-of-Custody" form and a laboratory assignment request form.

4.4 Field Analysis of Samples Using the XRF

The nine inch soil cores obtained from the test plots will be separated to 3" samples in the field and transported to a field laboratory for sample preparation and total lead analysis using XRF methods.

The individual tubes containing soil cores retrieved from a depth of 0-9" inches will be separated in the field into 0-3", 3"-6" and 6"-9" inch sections ("A", "B" and "C" respectively) as described in section 4.2.2. These separate sections will be capped and stored for subsequent XRF analysis.

4.4.1 XRF Field Laboratory

A field laboratory will be established for sample preparation and analysis. Access into the working area will be limited to NTH personnel during sample preparation and analysis.

During sample preparation and equipment decontamination, proper personal protection, including latex gloves and a dust mask, must be worn. Personnel not wearing the proper personal protection equipment may not enter the room during sample preparation.

To insure proper chain-of-custody of the samples and instrumentation, the working area must be capable of being securely locked when representatives of NTH are not present. Equipment used during sample preparation and analysis must be kept in a clean, covered container.

4.4.2 Instrument Calibration

A calibration curve for the Oxford Lab-X 1000 XRF instrument has been developed to quantify the concentration of lead in soil. The calibration curve was developed using samples of accurately known lead concentrations along with the XRF unit's built-in programming. The calibration program is stored in the instrument's memory. During the course of the investigation, the calibration curve will be revised, as required, to reflect site specific conditions.

XRF analytical methodology is dependent on the matrix of the specific soil type and characteristics of the lead being analyzed for. For this reason, a site specific calibration curve will be developed and utilized for the Granite City Pilot Study.

Selected soil samples will be split during the initial sampling. Half the sample will be analyzed for total lead by Martel Laboratories, while the remaining sample will be archived for later use as a calibration standard. Soil samples will be selected to provide a range of soil lead concentrations representative of conditions at the project location.

4.4.3 Sample Preparation

The fundamental principle of XRF is the detection and measurement of X-rays emitted from an excited atom. The most important consideration in the measurement of emitted X-ray energy is the influence of sample matrix effects. Soil samples are typically heterogeneous. Natural variations in such factors as particle size and moisture content, as well as the heterogeneous nature of the lead concentration, can result in significant accuracy problems if not corrected. Sample matrix effects can never be fully eliminated; however, through proper sample preparation and calibration techniques, they can become relatively insignificant.

In order to maintain analytical accuracy, quality assurance and quality control, the sample preparation procedures have been adopted from the following references:

> "The Chemical Technicians Ready Reference Handbook", 1990 Annual Book of ASTM Standards, Volume 4.08, and
>
> "Test Methods for Evaluating Solid Waste", Volume 1A, SW-846, Method 7000 Atomic Absorption Methods, September 1986.

1. **Use clean sample preparation area.** The sample preparation area must be clean. Decontaminate all sample preparation equipment according to the following procedures:

   a. The equipment is washed and scrubbed with a brush using distilled water and Alconox detergent solution to remove any soil residue.

   b. Rinse the equipment with distilled water and dry with paper towels.

   c. Place the decontaminated equipment in a clean, lidded container.

2. **Remove coarse material and sieve.** Weigh sample and record sample weight. Place a number 10 mesh sieve overtop a drying plate. Empty entire contents of sample bag into the sieve. Remove coarse material, foreign objects such as feathers and asphalt and any visible organic materials. Work soils through sieve as needed (wearing latex gloves and dust mask). Describe soil type and record information in logbook. Weigh and record the soil fraction passing through the number 10 mesh sieve.

3. **Dry sample.** Place the drying plate containing sieved soils into the microwave. Turn the oven on for 3 minutes. If the microwave has variable energy levels, use at the highest level. After the set time has elapsed, remove the plate and soil, allow to cool and determine and record the weight. Mix the soil carefully and reheat the sample at one minute intervals, until mass determinations have an insignificant affect on the calculated moisture content. A change of 0.1% or less of the initial wet mass of the soil should be acceptable for most samples. Record the amount of drying time needed. This procedure for drying soils was taken from ASTM Standard Test Method D4643-87.

4. **Homogenize sample.** Place the dried sample on a clean sheet of paper. Thoroughly homogenize the sample with a spatula using the cone and quartering procedure presented in the Second Edition of the Chemical Technicians' Ready Reference Handbook.

5. **Place sample in XRF cup.** After quartering the sample, select two opposite quarters and fill the XRF sample cup to the fill line. Use the remaining 2 quarters for flame AA analysis when appropriate.

6. **Archive sample.** When the XRF analysis is completed, archive the sample in a labeled sample container. The quartered

material not tested should be archived along with the tested sample.

4.4.4 Operating Procedures

The Oxford Lab-X 1000 XRF instrument is equipped with a menu system to perform the different options available for the unit. Options are chosen using the numbered key pad.

A detector resolution check of the XRF unit must be performed not less than once a week. The resolution check is a 20 second measurement of the unit's built-in standards. The value from the resolution check gives an indication of the condition of the detector. In addition, the electronic gain or Digital to Analog Converting (D.A.C.) value of the unit is determined. The resolution value and D.A.C. value must be recorded after each test. The resolution and D.A.C. value are recorded on the XRF Detector Resolution Log, Figure 8.

The resolution value must be less than 32 percent and the D.A.C. value must be above 1,500 and typically ranges between 1,500 to 3,500 volts/volts. If the resolution value or D.A.C. is not acceptable, Oxford Analytical, Inc. must be notified immediately to repair the equipment.

4.4.5 Quality Assurance for XRF Data

The Lab-X 1000 has a printer that displays the results of the analysis, including the date, calibration being used, reported concentration, and a message if the resultant lead concentration exceeds preset target levels.

For quality control purposes, the original printout from the instrument will be preserved in a binder. In addition, an XRF data log will be completed for each analysis. A sample XRF data log is shown as Figure 9, XRF Data Log. The data will not be considered validated unless a cross check has been made between the printout and the log.

To evaluate the reproducibility of the XRF results, 20 percent (1 in 5) of the samples will be reanalyzed using XRF without disturbing or handling the prepared sample.

The accuracy of the XRF measurements will be evaluated by submitting random samples to the selected laboratory for analysis. Ten percent (1 in 10) of the samples analyzed using XRF will be split during sample preparation and sent to the selected laboratory for total lead analysis (EPA Method 7420).

Data obtained from the tests for reproducibility will be evaluated by computing the "I" statistic for sets of samples. The "I" statistic is computed using the following procedure:

The absolute value of $$\frac{(\text{Sample A} - \text{Sample B})}{(\text{Sample A} + \text{Sample B})} = I$$

Sample A and Sample B are consecutive tests or splits of the same soil sample.

An out-of-control situation will occur when "I" is greater than 0.2. When an out-of-control is detected, efforts will be made to determine the cause, and corrective actions will be taken to bring the process under control. Corrective actions taken will be fully documented. Further analysis will not continue until the process is determined to be under control.

5.0 BENCH TOP STUDY

A bench top study will be conducted using composite soil samples from five (5) selected test plots. Physical, chemical and agronomical tests will be performed to classify the soils at this site and determine the optimum dosages of lime and nutrients. Lime will be applied to reduce the mobility of lead in the soils and nutrients will be added for a healthy growth of plants and grass.

5.1 Sample Collection and Preparation

The bench top study will be coordinated with the field sampling effort (see Section 4.0). During the initial sampling phase, five (5) composite soil samples will be collected for the bench top study. From the sixteen test plots used in the pilot study, 5 test plots will be selected and identified for the collection of the composite soil samples. The exact plots to be sampled will be selected by NTH personnel based upon lead levels encountered in the field. Generally, test plots with the highest and greatest variability of lead levels will be selected in order to model the anticipated worst case scenario.

The 5 composite soil samples will be obtained from the same location on three occasions: (1) prior to homogenization, (2) after the soil has been homogenized prior to the addition of lime and nutrients and (3) after a period of nine months/one year.

The 5 composite soil samples will be prepared by sampling eight separate locations within each of the 5 selected test plots. In each selected test plot approximately 8 ounces of soil will be collected at each sampling grid point. The depths of the material collection for composite samples will range from the surface to nine inches at each sampling point as discussed in Section 4.3. Each jar will be carefully marked as to test plot, sample number, date and personal ID. All sample jars will be shipped to the NTH laboratory.

Upon arrival at NTH's laboratory, the 8 ounce samples from each sampling location in each test plot will be mixed to develop one representative composite sample for each selected test plot. Once mixing is complete, composite samples will be tested to determine the physical, chemical and agronomical characteristics of the soil from each test plot.

5.2 Laboratory Analysis

During the initial sampling, prior to homogenization, physical, chemical and agronomical tests will be performed on each composite sample to characterize the material. Results of these laboratory tests and engineering analysis will be used to develop a qualitative and quantitative assessment of nutrients and seed mix for grass growth and lime additive to reduce lead mobility.

After the soils are homogenized, five composite samples will be obtained for chemical testing. The results of these tests will be used to determine the mobility of lead in the homogenized soil prior to the addition of lime or nutrients.

One year after the homogenization and treatment of the test plots is complete, additional samples will be collected to evaluate the grass growth and whether or not there have been any changes in lead mobility for each test plot area. Laboratory analysis will focus on chemical testing of the soil samples.

5.2.1 Physical Testing

Physical testing will be performed by NTH in their Farmington Hills laboratory. The following tests will be performed on the five composite soil samples collected during the initial sampling event:

- grain size (ASTM D422); and
- plasticity (ASTM D4318).

5.2.2 Chemical Testing

Chemical testing will be subcontracted to an analytical laboratory. The following chemical tests will be performed on the five composite soil samples collected during the three sampling events:

- soil pH (ASTM D4972);
- total lead content (Method 7420, EPA SW846); and
- lead leachability by TCLP analysis for metals (Method 1311, EPA SW846).

TCLP will be performed prior to and after the addition of lime.

5.2.3 Agronomical Testing

A soil fertility analysis will be performed on the five composite samples collected during the initial sampling period. Based on the results of this study a recommendation will be made for the appropriate dosage of lime, nutrients and a seed mixture.

The soil fertility analysis and lime recommendation will be based on the procedures used in the Soil Testing Laboratory at Michigan State University. These procedures are the standard ones used in such laboratories in the North Central Region of the United States. These procedures are used as the basis for fertilizer recommendations for a wide range of plants including grasses usually used for home lawns, golf courses and athletic fields. The reference is as follows:

Dahnke, W.C. 1988. Recommended chemical soil test procedures for the North Central Region. North Central Reg. Pub. No. 221 and Dak. Agric. Exp. Sta. Bull. 499.

5.3 Lime and Nutrient Optimization

A sixteen-ounce jar of each laboratory composite sample will be sent to Michigan State University (MSU) for analysis of soil fertility and a maximum pH which the soil can sustain. Based upon laboratory analysis of the five composite samples, MSU will recommend a grass seed mix, identify the optimal lime dosage to achieve the maximum sustainable pH and determine the nutrient requirements to promote maximum vegetative growth. Donald R. Christensen, Ph.D., acting as a consultant to NTH, will be directing this work at the Soil Science Department of MSU.

5.3.1 Lime Optimization

Lime addition to acidic soils is a common agricultural practice. In general, a pH of 6.5 to 7.0 is the goal of liming agricultural soils. However, for this study, elevated pH values will be considered as a means of reducing lead mobility in the soils.

According to an article on In-Place Precipitation and Immobilization published in Hazardous Waste & Hazardous Materials, 1988, a pH of 8.5$\pm$ minimizes lead mobility in soil.

MSU will determine the maximum pH level which can support good vegetative growth in the collected soil samples. Soil pH will be determined using a 1:1 soil to water volume ratio. The pH of the water-soil mixture will determined after allowing 10 minutes for the mixture to equilibrate. The Shoemaker, McLean & Pratt (SMP) buffer method will be used to determine the liming requirement. The SMP method involves adding a standard buffer solution to the soil sample, allowing the mixture to equilibrate for 30 minutes, determining the pH of the SMP-soil solution and relating this pH to a calibration curve to obtain the liming requirements. Based upon these test procedures, MSU will make a liming recommendation for each of the composite soil samples from the five test plots. This recommendation will be in pounds of 100 percent pure calcium carbonate per acre.

The recommended quantity of calcium carbonate will be applied to the test plot soil in the field by NTH personnel after homogenization. The purity of the liming material will be considered when calculating the amount applied according to standard agricultural practice. For example, if 2000 pounds per acre of 100 percent pure lime is recommended, 2500 pounds per acre of 80 percent pure liming material would be an acceptable substitute.

Fineness of the liming material will also be considered prior to application. Finer liming materials react with the soil much quicker than coarse liming materials. The liming materials used will meet typical minimum fineness requirements for agricultural purposes. Based upon the recommendation of MSU, more stringent fineness requirements may be adopted.

5.3.2 Nutrient Optimization

Nutrients are essential to vegetative growth. The nutrients that grasses utilize for growth may come from many sources, including soil reserves and fertilizer additions. Nitrogen, phosphorous and potassium are typically required in large quantities while nutrients such as magnesium and calcium are required in smaller quantities.

The Soil Science Department of MSU will determine the available nitrogen, phosphorus, potassium, calcium and magnesium in the soil. Levels of plant available nutrients in the soil will be determined by atomic absorption spectrometry or spectrophotometer. Based upon the selected grass type and levels of nutrients present in the soil, MSU will prepare a fertilizer recommendation.

5.4 Field Application

Once the optimal lime and nutrient application rates have been determined, homogenization of test plot areas will be performed. Section 6.0 fully describes the homogenization operation. Composite samples will be obtained at this time to determine total lead concentration, pH and the mobility of lead in the soil. After the composite samples are obtained, lime and nutrients will be applied.

Approximately nine months/one year after homogenization and treatment is complete, five composite samples will again be collected from the plots initially tested for soil characterization purposes. Samples will be collected according to the same procedures used during the initial composite sampling program and at the same locations. The one year period is established to allow for reaction between the soil and lime and nutrient additions.

These soil samples will be tested to assess the effects of the homogenization and lime addition operation on lead mobility. The results of these tests will be compared to the results obtained on the soil samples collected during the initial sampling round. From these results, NTH will develop a qualitative and quantitative assessment of lead mobility.

6.0 HOMOGENIZATION AND TREATMENT OF SOILS

A field operation will be conducted to evaluate the effectiveness of homogenizing and treating near-surface soils. This portion of the project will determine if full-scale implementation of this technology is a practical remedial technique. The field operation will involve homogenization of the near-surface soils followed by a site specific treatment using lime and nutrients. As part of this field operation, the equipment used to homogenize the soils will be evaluated along with the effect of applying lime and nutrients to treat the homogenized soils.

As stated, a total of sixteen representative test plots will be selected for homogenization and treatment. In general, it is anticipated that lead concentrations for the selected plots will range from ± 500 mg/kg to ± 2000 mg/kg. The areas selected will be representative of the wide variety of conditions expected during a full-scale operation. Two plots with replicate soil concentrations will be selected for each range of lead concentrations to analyze the tilling/mixing equipment utilized during the homogenization and treatment field operation.

6.1 Tilling/Mixing Equipment

The first issue addressed as part of the field operation is the evaluation of the tilling/mixing equipment to be used during homogenization of the surface soils. Two separate types of tilling/mixing equipment will be selected for the study; hand-operated, self-propelled tillers and small tractor-driven tillers.

Of the sixteen test plots used during this study, eight (8) plots will be homogenized using hand-operated tillers and the other eight plots will be homogenized using small tractor-driven tillers as shown in Figure 1. Homogenization of the soils in each plot will be performed according to the procedure described in Section 6.2. NTH will evaluate both types of soil tilling/mixing equipment to determine their applicability and effectiveness in homogenizing surface soils. Evaluation criteria will include:

- Time to complete a mixing pass;
- Ability to control mixing depth;
- Time to homogenize the test plot;
- Tilling/mixing effectiveness;
- Maneuverability; and
- Dust control.

6.2 Field Operation

The field work during this pilot study will involve two distinct phases; homogenization of the near-surface soils and application of lime and nutrients. The following sections discuss both operations.

6.2.1 Homogenization

The primary focus of the field operation is to determine the efficiency and effectiveness of soil homogenization. Since the test plots will be selected based upon lead in the surface soils, efficiency will be evaluated based upon the final distribution of lead after homogenization of the soils to a depth of nine inches. The number of passes and type of tilling/mixing equipment will be two physical parameters studied while the variability of the lead content within the test plots will be the chemical parameter monitored. Each test plot will be homogenized vertically from 0-9". Homogenization of soil will continue, in the form of equipment passes, until field tests indicate that the lead distribution has approached a constant. The pass number, samples obtained, and comments will be recorded on the Field Sampling Form, Figure 10.

Four of the selected plots will be tested to determine the optimum tilling/mixing effort for homogenization. Initially, two to three passes may be required to meet the required tilling depth of nine inches. Adjustments will be made, as required, during the field operation.

The degree of homogenization will be analyzed in the field by computing the mean and standard deviation of lead concentrations using XRF test results from samples collected after each pass. Homogenization will continue until the mean and standard deviations from consecutive passes approach a constant. A data curve similar to Figure 11, Plot of Mean Soil Lead Concentration Versus Number of Passes, will be used to determine the optimal number of equipment passes required to achieve relatively uniform mixing.

Once the optimal number of passes has been determined for homogenization, 24 samples will be collected and placed in 125 ml sample jars and shipped to Martel Laboratory Services, Inc. for analysis. Each jar will be clearly marked with project name and number, test plot, sample depth, date, time, and name of the field technician collecting the sample. Standard chain-of-custody procedures will be followed, as discussed in Section 4.0. The total lead content (EPA Method 7420) of each sample will be determined to assure accurate correlation with XRF results and to provide a more precise determination of lead concentrations and distribution throughout the test plot.

During the homogenization operation, air sampling will be performed to assure that ambient air quality standards are not exceeded. Personal air monitoring will also be used by personnel directly involved in the homogenization operations.

6.2.2 Soil Treatment Using Lime and Nutrients

Lime will be applied to eight (8) test plots after each plot has been homogenized. Nutrients will be applied to all sixteen (16) test plots. The quantities of lime and nutrients to be added will be based on recommendations provided by the Soil Science Department of Michigan State University, as detailed in Section 5.3.1. Lime purity and fineness will be considered according to standard agricultural practice. This will assure maximum effectiveness of the applied lime in terms of neutralizing soil acidity and reaction with lead compounds. Nutrient additions, such as nitrogen, phosphorous, potassium, magnesium and calcium, will also meet MSU's recommendations as described in Section 5.3.2. Nutrients will be applied to the test plots prior to seeding.

6.2.3 Seeding and Maintenance

Based on the recommendation of the Soil Science Department of Michigan State University, the recommended seed mixture will be applied to all of the test plots. After the test plots are seeded, a wheat/straw cover will be placed on the test plots to control erosion and retain moisture in the soil. At this time, a watering schedule will be established to maintain and support the growth of the grass on each test plot.

7.0 DATA MANAGEMENT

An adequate data management system is necessary to allow all personnel involved in the project easy entry and access to data for validation, interpretation, analysis and decision making purposes. The following sections describe the procedures that will be used in documenting, tracking, organizing, and presenting all data collected during the Granite City project to ensure they are of sufficient quality to provide an accurate site characterization.

7.1 Field Activity Documentation

Documentation of activities is important to provide an accurate account of work performed in the field during this study. The following methods will be used to record data:

7.1.1 Daily Logbooks

The field supervisor will be responsible for maintaining a daily logbook and sample collection documentation for field activities. Information from individual field personnel, who have been designated to perform specific tasks, will be collected and maintained by the field supervisor. Observations made by individual field personnel will be promptly recorded in the logbook. Results from laboratory or field equipment calibrations will also be recorded in the logbook. The original records will be retained for the project files.

Logbook entries will contain accurate descriptions and documentation of an individual's field activities. Logbooks will form the basis for the written reports, therefore, they will contain only facts and observations. Entries will be signed and dated by the individual making the entry on a daily basis.

7.1.2 Record Validation

Before the release of sampling documentation, project personnel will assemble all records and cross-check the information on corresponding sample labels, sample data sheets, custody records, logbooks, and other accountable documents to ensure that data pertaining to each particular sample is complete and consistent throughout the record. Examples of the sample labels, sample data sheets and NTH chain-of-custody are provided as Figures 3, 4 and 5.

If an error is made on an accountable document assigned to an individual, the individual will make corrections by crossing a line through the error and entering the correct information. Any subsequent error discovered on an accountable document will be corrected by the person who made the entry. All corrections will be initialed and dated.

7.1.3 Photographs

When photographs are taken, they will be numbered to correspond to logbook entries. The name of the photographer, date, time, location and description will be entered sequentially in the logbook as photos are taken. Each undeveloped roll of film will be properly marked to identify the photographs that will be developed. Once the photographs have been developed, they will be numbered corresponding to the logbook descriptions and labeled accordingly. Negatives will also be numbered, labeled, and stored in a secure location.

7.2 Data Base Management

Information collected from each sample location will be recorded using a data management computer software package such as dBASE IV. The following information will be maintained for each sample location:

- sample identification number (includes sample area, sample location, date sampled and sampler)
- sample location
- sample depths
- ground surface elevation (if available)
- analytical results for each sample

Analytical results will include XRF, Flame AA and quality control analyses (i.e. duplicate samples) when appropriate.

7.2.1 Laboratory Documentation

Laboratory generated analytical data will be given to the personnel responsible for data entry. They are responsible for making copies of the data and filing these copies along with the appropriate chain-of-custody form in the shipment logbook discussed in Section 4.0. The data will then be entered into the data base management system. The original analytical data sheets received from the laboratory will be filed in binders chronologically by the date data are received to minimize handling and loss of results.

7.2.2 Data Entry Validation

The data base will be updated daily with information from the field office. Daily entries will be printed on backup hard copy and cross checked by a second party other than the data entry

7.1.3 Photographs

When photographs are taken, they will be numbered to correspond to logbook entries. The name of the photographer, date, time, location and description will be entered sequentially in the logbook as photos are taken. Each undeveloped roll of film will be properly marked to identify the photographs that will be developed. Once the photographs have been developed, they will be numbered corresponding to the logbook descriptions and labeled accordingly. Negatives will also be numbered, labeled, and stored in a secure location.

7.2 Data Base Management

Information collected from each sample location will be recorded using a data management computer software package such as dBASE IV. The following information will be maintained for each sample location:

personnel. A backup copy of the data base will also be maintained on a floppy disk. Record of dates when the backup programs were made will also be maintained. The tracking form will outline the date on which an action was performed to the data base and the person responsible for that action.

At the completion of each sampling round and subsequent sample analysis, a quality control review process will be conducted on the data base. Quality control measures will consist of a second and third party review of the data for typographical and technical errors. The review process will be completed by a review and final approval from the project manager.

7.2.3 Management of XRF Samples During Homogenization Operation

During the homogenization operations soil samples will be collected after each tilling/mixing pass. These samples will be analyzed for total lead using XRF methods. The data base will include the sample plot number, sample identification number, sample collection date, lead concentrations and sample location coordinates.

Data entry will be displayed on a printout and verified for accuracy by personnel other than data entry personnel. Once validated the data will be used by the field supervisor to determine the optimal number of tilling passes as described in Section 6.2.

7.2.4 Management of Air Monitoring Data

A database management system will also be utilized to accommodate the air quality data accumulated during the Granite City pilot study. The data base will be developed to include the date the sample was collected, sample plot location, airborne lead particulate concentration, location coordinates for each hi-vol sampler, and the wind speed and direction. This will allow interaction between the hi-vol data and soil lead data.

Air quality data collected during the Granite City project will be entered into the data base, displayed on a printout and verified for accuracy by personnel other than data entry personnel. The advantages of the database management system include easy access by project personnel, the processing of the data to produce graphical displays, summary statistics, and other analyses that can be used in evaluating project data.

8.0 DATA ANALYSIS AND INTERPRETATION

The objectives of the sample data analysis and interpretation to evaluate tilling/mixing efficiencies (i.e. homogenization and redistribution of lead) and to characterize the effect that homogenization has on surface soil lead concentrations. The tilling/mixing efficiency will be evaluated by performing statistical analyses on the measured lead concentrations within the selected plots under various conditions. A soil lead concentration reduction curve will be developed.

8.1 Soil Data Presentation

All soil samples collected during the Granite City project will be located on a site grid. The sample locations will then be plotted on a detailed map of the area with the analytical results displayed adjacent to each sampling location. This visual display will aid in interpreting site conditions, identifying any data gaps or existing patterns, and if necessary, in selecting additional sampling locations.

The raw data will also be graphed and tabulated. The data tables will include sample designations, depth of samples, and chemical concentrations. To perform the data analysis and quality control, soil lead data will be down-loaded from the data base management system into various commercially available statistical software programs. Examples of these programs include Lotus 123, Windows Excel or STATGRAPHICS.

8.2 Data Distribution

An understanding of the underlying distribution of the data is necessary in order choose applicable statistical methods. The most commonly used methods require that the data must fit a normal distribution. Therefore, the data will be tested for normality using appropriate techniques. If the data are found not to be normally distributed, a mathematical transformation will be applied to the data so that they will approximately conform to normality.

8.3 Tilling/Mixing Optimization

Four of the selected plots will be tested to determine the optimum tilling/mixing effort for homogenization. For each of these plots, the soil lead concentration will be measured between passes. Soil samples will be collected as discussed in Section 4.0.

For each of the optimization plots, the mean and standard deviation of the lead concentration after each tilling/mixing pass will be calculated. A graph of the mean surface (0-3") soil lead concentration versus the number of passes will then be constructed.

An example of such a graph is shown on Figure 11. Additional graphs of the standard deviations versus the number of passes will also be constructed.

Tilling/mixing will continue for each optimization plot until the mean and standard deviation of lead concentrations from consecutive passes approach constants. The graphs and other statistical analyses will then be used to analyze the effect the number of passes has on the mean surface soil lead concentration. Based on the observed behavior, the optimal number of tilling/mixing passes required to achieve relatively soil homogeneity will be determined.

8.4 Regression Analysis

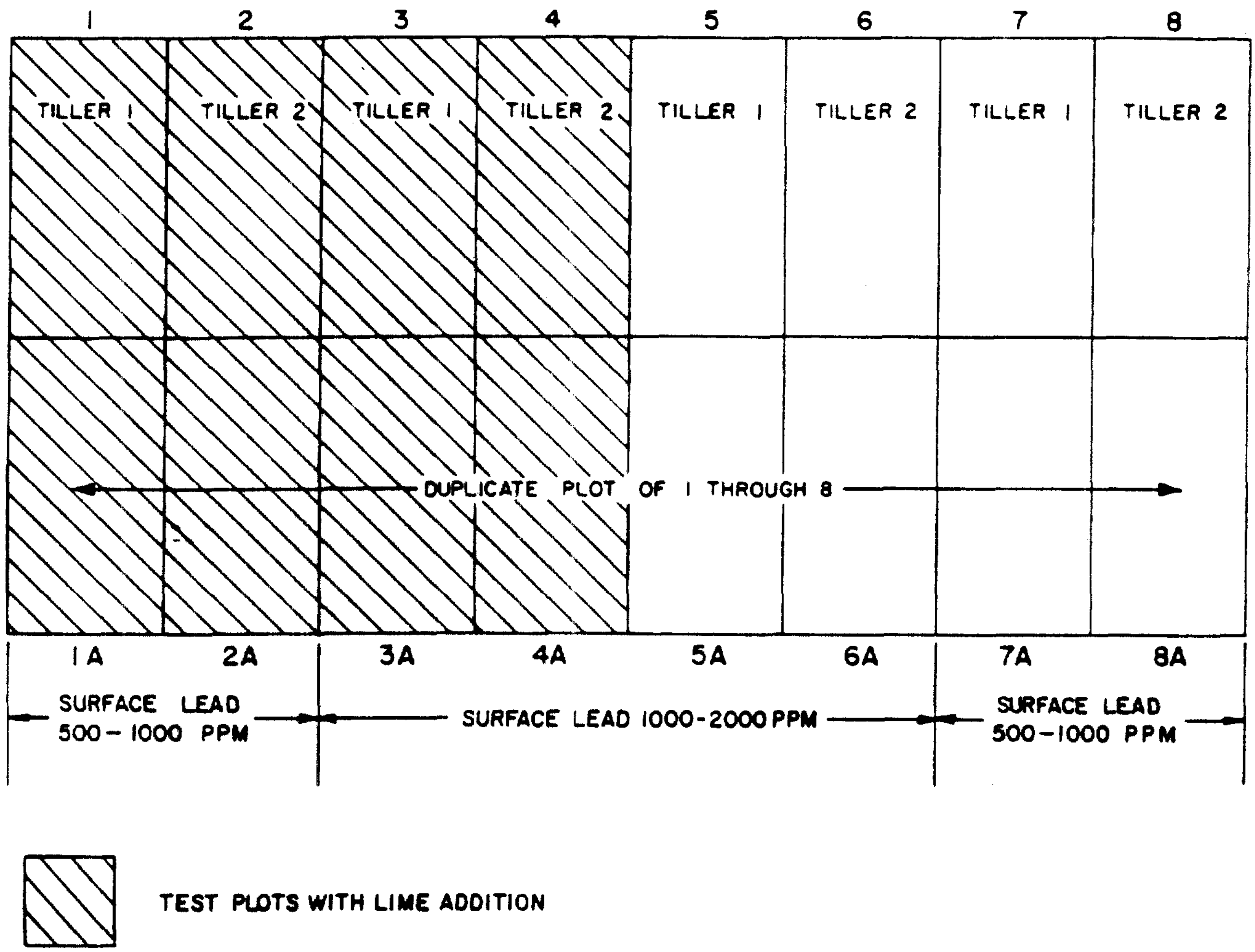
Multiple regression techniques will be used to develop a relationship between the mean surface soil lead concentrations before homogenization to the mean surface soil lead concentrations after homogenization. The regression analysis will consider each of the variables described in Section 3.2:

- Homogenization/mixing method
- Initial soil lead concentration
- Lime addition (9 months/1 year later)

Figure 12, Regression of Final Lead Concentrations Versus Initial Surface Lead Concentrations, shows an example of a regression curve using one of these variables.

The relationship developed as a result of the regression analysis will be used to predict the effect of homogenization for various initial surface soil lead levels. The regression will also be used to establish the effectiveness of the different tilling/mixing methods and lime addition. If possible, the relationship will be expressed in the form of a concentration-reduction curve which will also display the ranges of expected results at different levels of confidence (e.g. 95, 99, or 99.5%).

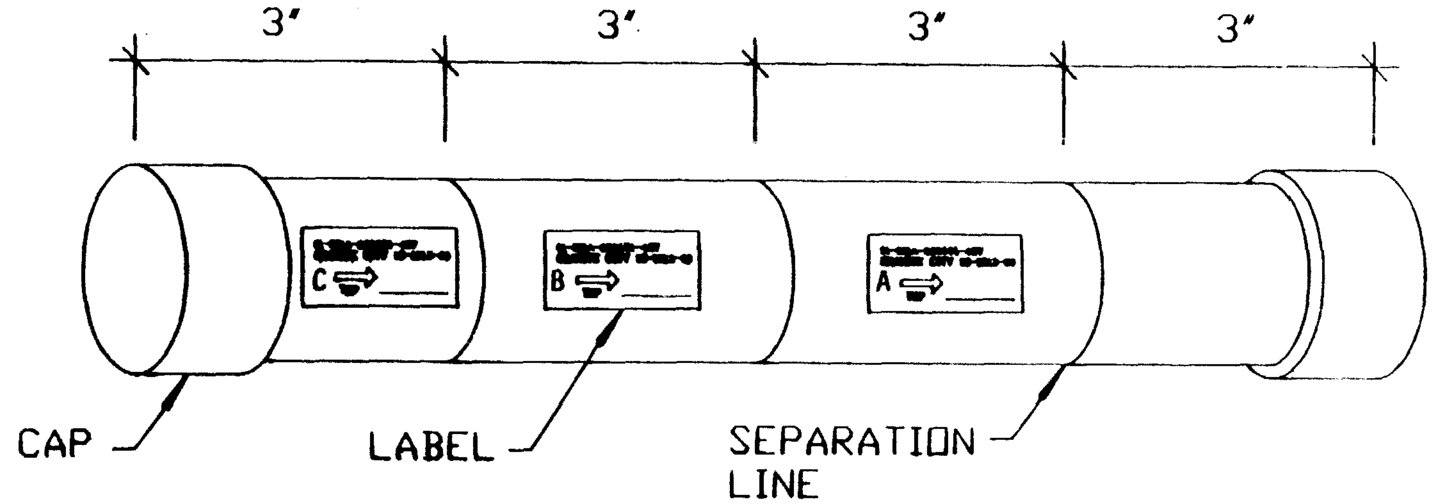
LIST OF FIGURES

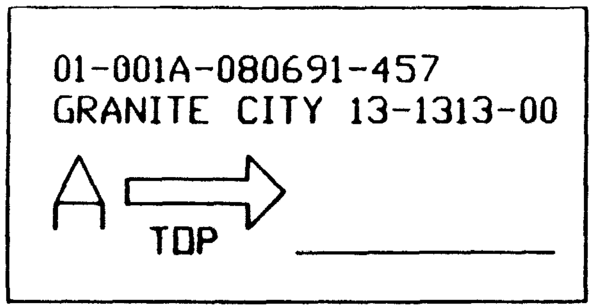


NOTE:

- BASED ON THREE VARIABLES; SURFACE SOIL LEAD CONCENTRATIONS, LIME ADDITION AND TILLING METHOD
- ALL TEST PLOTS WILL HAVE NUTRIENTS APPLIED

Scale: NONE	**PLOT SELECTION CRITERIA** GRANITE CITY MADISON COUNTY, ILLINOIS
Date: 9/24/91	
Drawn: S.F.D.	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services 860 SPRINGDALE DRIVE, EXTON, PENNSYLVANIA 19341 (215) 524-2300
Checked:	Project No.: 13-1313-00 — FIGURE 1





1. OBTAIN SAMPLE
2. REMOVE LINER AND INSPECT RECOVERY
3. AFFIX LABELS IN PROPER POSITIONS
4. SEPARATE LINER INTO 0-3", 3"-6", AND 6"-9" SECTIONS (A,B,C)
5. RECAP SEPARATED SECTIONS
6. TRANSPORT SAMPLES TO LAB AND STORE

Scale: N/A	SOIL CORE/LABEL EXAMPLE
Date: 9-24-91	
Drawn:	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services
Checked: VB.F.	Project No.: 13-1313-00 FIGURE 3

NTH Consultants, Ltd.
A Neyer, Tiseo & Hindo Company
38955 Hills Tech Drive, Farmington Hills, Michigan 48331-3432 • (313) 553-6300 • Fax (313) 489-0727

PROJ. NO.	PROJECT NAME:					NO OF CONTAINERS	ANALYSIS TYPE REQUESTED							
	PROJECT PROFESSIONAL:													
SAMPLERS: (Signature)														
I.D. NO.	DATE	TIME	COMP	GRAB	STATION & LOCATION									REMARKS FIELD PARAMETERS

LABORATORY:

Relinquished by:	Date	Time	Received by:	Relinquished by:	Date	Time	Received by
Relinquished by:	Date	Time	Received by:	Relinquished by:	Date	Time	Received by
Relinquished by:	Date	Time	Received by laboratory:	NOTE: ANALYTICAL LABORATORY SHALL SUBMIT QA/QC DATA WITH ALL CHEMICAL TESTING REPORTS. Data to include: Blanks, Duplicates, Recoveries, and parameter control limits. Please calculate duplicate and recovery data to reflect control limits.			

Scale: N/A	CHAIN OF CUSTODY RECORD FORM
Date:	
Drawn:	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services
Checked: DB 7	
Project No.: 13-1313-00	FIGURE 5

SAMPLE DATA SHEET

Soil ☐ Sediment ☐ Surface Water ☐ Other ____________

Sample ID Number: ____________ Split Sample Requested:

Sample Location: ____________ ☐ Yes ☐ No

____

Sample Depth: ____________

Sample Location Map:

Date: ____________
Time: ____________

Air Temperature: ____________
Weather: ____________

Sampler: ____________
Witness: ____________

Sampler: ____________
Witness: ____________

Quality Control Duplicate:

☐ Yes ☐ No

____

Description: ____________

____

Sample Location Morphology:
Flat, slope depression, gully,
hill top, Notes: ____________

____

Sample Stratigraphy: ____________

____

____

Trowel: ____________ Auger: ____________ Other: ____________

INFORMATION RECORDED BY ____________

Scale: N/A	SAMPLE DATA SHEET
Date:	
Drawn:	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services
Checked: DBJ	Project No.: 13-1313-00 — FIGURE 4

SAMPLE TRACKING LOG

SAMPLE ID	SAMPLING DATE	SHIPPING DATE	SAMPLE ID	SAMPLING DATE	SHIPPING DATE

Scale: N/A	SAMPLE TRACKING LOG
Date:	
Drawn:	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services
Checked: WB.7	Project No.: 13-1313-00 FIGURE 6

LABORATORY ASSIGNMENT REQUEST

DATE: ____________________

TO: ____________________

FROM: NTH Consultants Ltd.
38955 Hills Tech Drive
Farmington Hills, MI 48331

ATTENTION: ____________________

ATTENTION: ____________________

PROJECT NUMBER: ____________________

PROJECT LOCATION: ____________________

NUMBER OF SAMPLES: ____________________

SAMPLE IDENTIFICATION	TEST REQUIRED

NOTES: ____________________

The Above Request(s) Approved By: ____________________

	Scale: N/A	LABORATORY ASSIGNMENT REQUEST FORM
	Date:	
	Drawn:	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services
	Checked:	Project No.: 13-1313-00 — FIGURE 7

NTH Consultants, Ltd.
A Neyer, Tiseo & Hindo Company
38955 Hills Tech Drive, Farmington Hills, Michigan 48331-3432 • (313) 553-6300 • Fax (313) 489-0727

GRANITE CITY
13-1313-00

XRF Detector Resolution Log
Oxford Lab-X1000

DATE	HEAD NUMBER	RESOLUTION VALUE	D.A.C. VALUE

NOTE: If the resolution value is 32 **percent or greater contact Oxford Analytical, Inc. immediately.**

FIGURE 8

NTH CONSULTANTS, LTD.

GRANITE CITY
13-1313-00

XRF Data Log
Oxford Lab-X1000

DATE	SAMPLE ID	LEAD (ppm)	LEAD (ppm)	DATE	SAMPLE ID	LEAD (ppm)	LEAD (ppm)

FIGURE 9

NTH CONSULTANTS, LTD.

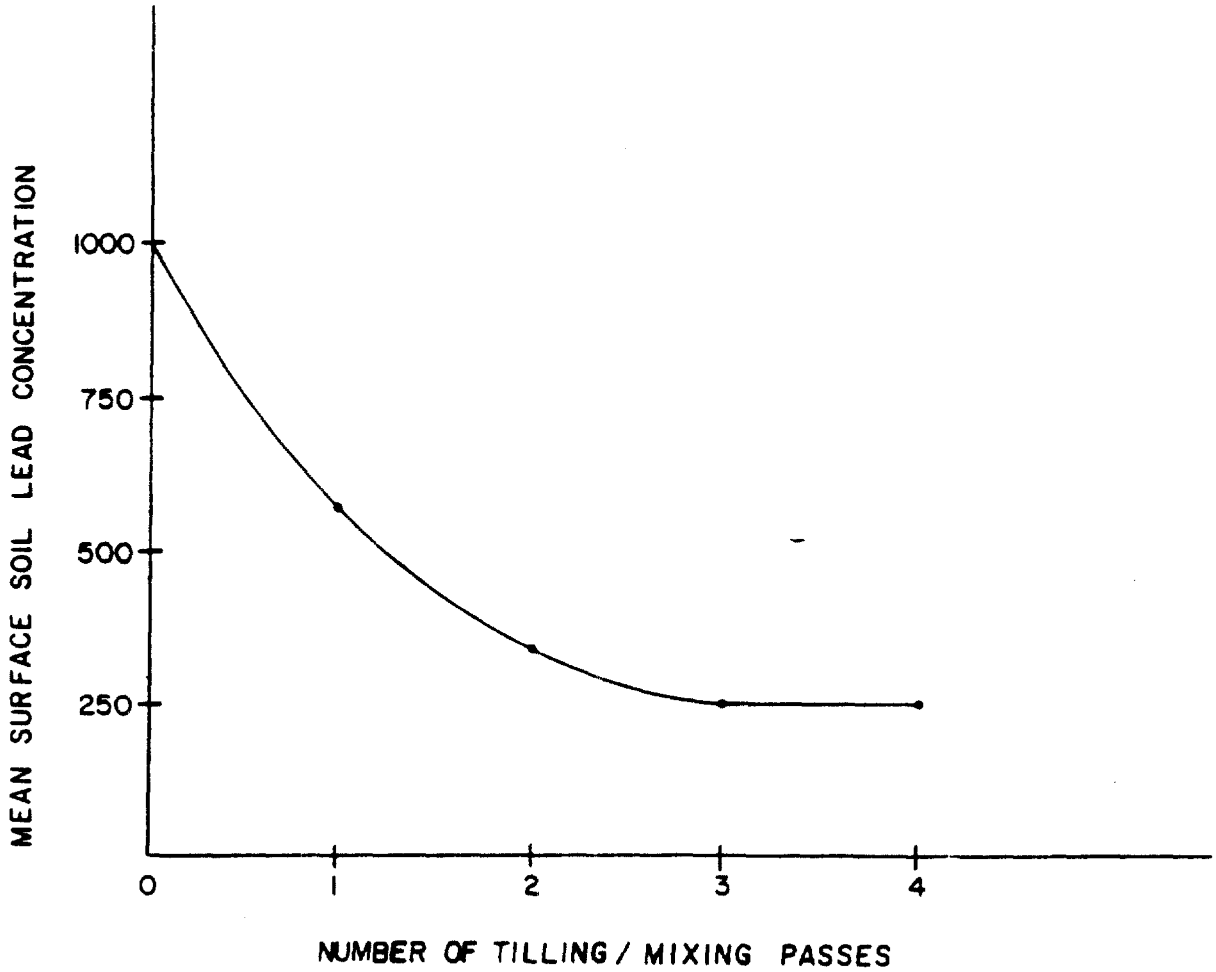
GRANITE CITY
SOIL HOMOGENIZATION/ TREATMENT PILOT STUDY
PROJECT NO. 13-1313-00

FIELD SAMPLING FORM
TILLIING/MIXING OPERATION

PLOT # ________
LOCATION: ______________________________
DATE: ______________
WEATHER: ______________
TEMPERATURE: ______________

PASS NUMBER	(✓) EMP. #	SAMPLE ID	COMMENTS
Start: Stop:			
Start: Stop:			
Start: Stop:			
Start: Stop:			

FIGURE 10

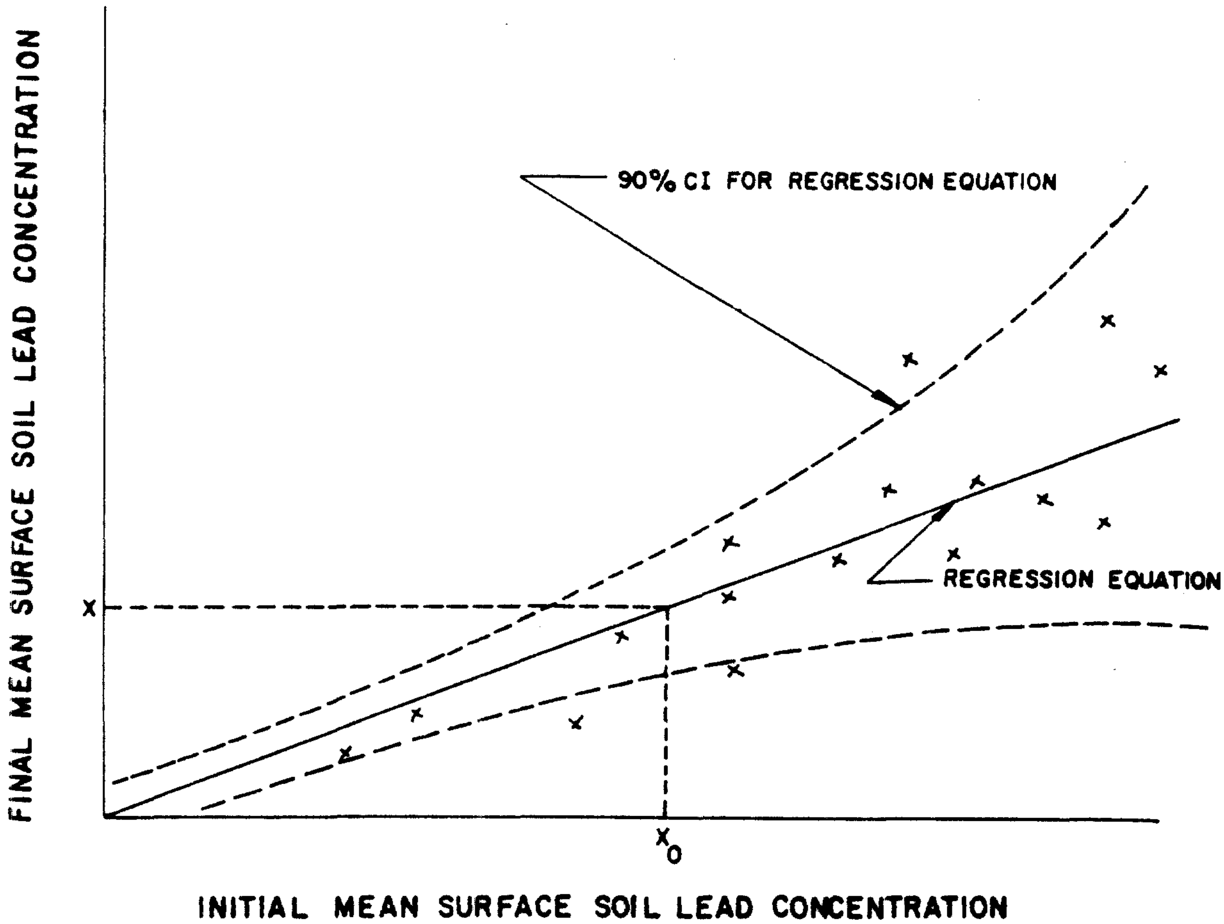


NOTE:
THE MEAN SURFACE SOIL LEAD CONCENTRATIONS ARE BASED ON THE 0"-3" SAMPLE INTERVAL FOR SIX LOCATIONS.

THE SOIL LEAD CONCENTRATIONS WILL BE MEASURED USING XRF INSTRUMENTATION.

EXAMPLE

Scale: NONE	PLOT OF MEAN SOIL LEAD CONCENTRATION VERSUS NUMBER OF PASSES GRANITE CITY MADISON COUNTY, ILLINOIS	
Date: 9/24/91		
Drawn: S.F.D.	NTH CONSULTANTS, LTD. Professional Engineering & Environmental Services 860 SPRINGDALE DRIVE, EXTON, PENNSYLVANIA 19341 (215) 524-2300	
Checked:	Project No.: 13-1313-00	FIGURE 11



NOTES:

- X IS THE CONCENTRATION THAT CAN BE ACHIEVED 90% OF THE TIME WHEN THE INITIAL CONCENTRATION IS X_0.
- REGRESSION IS DEVELOPED USING THE CONCENTRATIONS FROM THE SIXTEEN TEST PLOTS.
- FINAL MEAN SURFACE SOIL LEAD CONCENTRATION AFTER HOMOGENIZATION

EXAMPLE

REGRESSION OF FINAL LEAD CONCENTRATIONS VERSUS INITIAL LEAD CONCENTRATIONS
GRANITE CITY
MADISON COUNTY, ILLINOIS

NTH CONSULTANTS, LTD.
Professional Engineering & Environmental Services
Farmington Hills, Michigan

PROJECT NO:	DRAWN BY:	DATE:
13-1313-00	NAR	9-24-91
SCALE: NONE	CHECKED BY: GBJ	SHEET OF

FIGURE 12

APPENDIX A

PUBLIC HEALTH AND SAFETY MEASURES

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APPENDIX A

PUBLIC HEALTH AND SAFETY MEASURES

The public health and safety measures presented in this appendix have been developed specifically for the Soil Homogenization Pilot Study to be performed at test plots located in the vicinity of NL Industries/Taracorp Superfund Site in the City of Granite City, Illinois. The quality assurance/quality control procedures for the public health and safety measures are contained in Appendix C.

1.0 SITE INFORMATION

The NL Industries/Taracorp Superfund Site is located in the City of Granite City, Madison County, Illinois. Granite City, Illinois, is an eastern suburb of St. Louis, Missouri, and is located adjacent to the Mississippi River. Former operations at the site include metal refining and fabricating, secondary smelting (for the production of lead products) and battery recycling. The operations at the site, along with automotive emissions and other processes, may have contributed to elevated lead concentrations in the surface soils surrounding the NL Industries/Taracorp facility.

2.0 SCOPE OF WORK

In brief, the major tasks involved in the Pilot Study includes:

1) Selecting plots which are representative of elevated lead levels in soils.

2) Sampling soil in selected plots to establish levels of lead in the soil profile.

3) Homogenization of test plots to distribute the concentration of lead in surface soils.

4) Addition of lime to reduce the mobility of lead and addition of nutrients to enhance vegetative growth.

3.0 HAZARD EVALUATION AND CONTROL

3.1 Physical Hazards

If members of the public are permitted in the work area, the possibility of "struck by" injuries exists from hand tools or power equipment. Likewise, the possibility of a "striking" injury exists if someone should trip and fall on uneven ground or objects in the work area. The use of electrical equipment can present electrical hazards.

Only authorized personnel will be permitted in the work area. In addition, the test areas will be secured using a woven plastic fence staked around the perimeter of the work area during the tilling/mixing operation. The fence will be approximately three feet high and will serve as a preventative measure to restrict access to the test area. The fence will help maintain the integrity of the work area by preventing young children or animals direct access. The fence will be removed after grass is established.

The high volume air sampler will be operated on a three wire grounded circuit with ground fault interrupter protection.

3.2 Health Hazards

The most common route of exposure is the inhalation of dust containing lead. This route is also the most significant, as absorption through the lungs occur. Lead can also be ingested, but large amounts are needed to produce symptoms.

Public exposure to lead dust will be controlled by the measures contained in Sections 4.0 and 5.0 of this appendix.

4.0 TEST PLOT AIR MONITORING

4.1 Perimeter Air Sampling

Perimeter air samples will be collected throughout the duration of site activities. The purpose of this perimeter sampling will be three-fold:

* To monitor airborne lead levels for comparison with background levels.
* To document the levels, if any, of airborne dust caused during soil homogenization.
* To determine if any operational adjustments are necessary to reduce dust emissions.

4.1.1 Sampling Procedure

The Site H&S Coordinator will perform the perimeter air sampling for lead in accordance with the procedures of 40 CFR, Part 50, Reference Method for Determination of Suspended Particulate Matter in the Atmosphere (High Volume Method). The full sampling procedure will not be repeated here, but highlights follow.

The air samples will be collected from two stations established on the perimeter of the site. One location will be predominant upwind direction and one will be predominant downwind direction. Samples will be collected by drawing ambient air through a glass fiber filter at a rate of approximately 40 CFM. The sampler will be operated for a 24-hour period. The General Metal Works GMWS-2310 ACCU-VOL sampler will be used which automatically controls the sample flow rate to the set point. A continuous flow recorder will also be used as will an elapsed time indicator. Samplers will be automatically started and stopped by an electrical timer. The filter will be removed from the Hi-vol sampler, folded in half and inserted in a paper cartridge to protect the filter during transport to the laboratory for the analysis. The results will be compared to the EPA national ambient air quality standard for lead, 1.5 ug/m^3 arithmetic mean averaged over a calendar quarter.

4.1.2 Analytical Procedure

Sample analysis will be performed in accord with 40 CFR Part 50, Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air.

4.1.3 Meteorological Data

Meteorological measurements will be obtained using on site meteorological monitoring equipment. Wind speed and direction will be recorded on a continuous basis for the duration of the field activities. Ambient air temperature and precipitation will be recorded on a daily basis.

This meteorological data will be reduced into graphical and tabular formats and submitted in conjunction with the total suspended particulate and lead data.

5.0 DUST CONTROL

The laboratory analysis of Hi-vol air filters for lead outlined in Section 4.0 of this appendix, "Test Plot Air Monitoring", may require a week or more. As a result we will follow the procedure contained in Section 5.1, "Dust Monitoring During Homogenization", which will provide a real time (immediate) means to evaluate airborne particulate (total dust) using a hand held aerosol monitor (HAM). Total dust will be monitored since no method exists to measure lead in air in real time. This section provides a calculated action level for total dust based upon worst case of soil lead concentrations which may be encountered.

5.1 Monitoring During Homogenization

A HAM will be used to determine airborne dust concentrations to determine if dust control measures are required. Based on the maximum concentration of lead in soil at the test plot, an action level for total dust will be computed at which control measures will be initiated.

The action level is based on the following equation:

$$\text{Action Level} = \frac{(\text{EG mg/m}^3)}{(\text{Conc. g/g})(\text{Safety Factor})}$$

$$\text{Action Level} = \frac{(10^6\ \text{mg/kg})(\text{EG mg/m}^3)}{(\text{Conc. mg/kg})(\text{Safety Factor})} \qquad (1)$$

Where:

Action Level:	Concentration of total dust in the air at which control measures are required.
EG:	Exposure guideline of the contaminant of concern, e.g., its OSHA permissible exposure limit (PEL), OSHA action level or other health exposure guideline.
10^6:	Conversion factor -- 1 kg = 10^6 mg
Conc.:	Soil concentration of the contaminant of concern in mg/kg.
Safety Factor:	A factor between one and ten used to account for level of confidence.

EG (exposure guideline) is based on threshold limit value (TLV) for lead of the American Conference of Governmental Industrial Hygienists (ACGIH) -- 0.15 mg/m^3. The TLV assumes a forty hour per week exposure period and healthy workers. The TLV must first be adjusted for a twenty-four hour day, seven day per week exposure and adjusted with a safety factor of 100 to protect the general population. Thus, the adjustment factor by which the TLV is multiplied is calculated as is shown below. The EG (public) is calculated by multiplying the TLV by the adjustment factor. The EG (public) is substituted into the action level formula (equation 1).

$$\text{Adjustment Factor} = \frac{\text{40 hours per week}}{\text{24 hours per day} \times \text{7 days per week} \times 100} \qquad (2)$$

$$\text{EG (public)} = \text{(TLV) (Adjustment Factor)} \qquad (3)$$

EXAMPLE:

The action level for a lead in soil concentration of 2000 mg/kg is calculated as appears below. Concerning the selection of the safety factor in equation 1, the confidence level is high because the high end of the range of lead in soil concentrations (2000 mg/kg) is used and because the safety factor in equation 2 is large (100). Thus, a safety factor of 2 is selected for equation 1.

Adjustment Factor = 0.00238 (4)

EG (public) = 0.000357 mg/m^3 (5)

$$\text{Action Level} = \frac{(10^6\ mg/kg)\ (0.000357\ mg/m^3)}{(2000\ mg/kg)\ (2)} \tag{6}$$

Action Level = 0.0893 mg/m^3 = 89.3 ug/m^3

The action level for the public will be measured in perimeter of the work area where a potential for community exposure may exist.

5.2 Control Measures

5.2.1 During Homogenization

If two consecutive measurements taken five minutes apart with the HAM show dust levels in excess of the action level, the work will be slowed down or work will stop for five minutes. If excessive dust levels persist, water will be applied to the work area. These control measures are in addition to the control measures outlined in the following section.

5.2.2 Post-Homogenization

As a standard operating procedure, a dust control blanket will be used to cover soil in the test area after homogenization. The dust control blanket will help retain moisture in the soil particles and decrease the potential for public exposure to the homogenized soils. The dust control blanket will be used throughout the pilot study for ground cover to prevent dust and to promote grass seed germination and grass growth after the homogenization is completed.

The dust control blanket is made of wheat straw with light, photodegradable netting on one side and is sewn together with biodegradable cotton thread. After a period of 60 - 90 days, the netting will disintegrate. The wheat straw will serve as a cover to control dust and will eventually decompose.

APPENDIX B

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

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APPENDIX B

QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

1.0 INTRODUCTION - SOIL SAMPLING ONLY

1.1 Purpose

The purpose of the Quality Assurance/Quality Control (QA/QC) procedures are to produce reliable data of known and documented quality generated throughout the study by:

- Ensuring the validity and integrity of the data;
- Assuring and providing the mechanisms for on-going control of data quality;
- Evaluating data quality in terms of precision, accuracy, reproducibility and data capture (recovery); and
- Providing usable, quantitative data for analysis, interpretation and decision-making.

1.2 Scope

The following sections describe the methods for the QA/QC that will be utilized to enhance data quality for the Soil Homogenization Pilot Study in Granite City, Illinois.

The Quality Assurance/Quality Control (QA/QC) procedures to be used for both field operations and laboratory analysis are summarized below.

1.2.1 Field QA/QC methods include:

- Sample Custody (sample documentation, logging, and shipment);
- Field Audit Procedures (if necessary);
- Field Measures of QA/QC (duplicate samples);
- Field Sampling Procedures; and
- Field Equipment Calibration, Operation, and Maintenance (standard operating procedures).

1.2.2 Laboratory QA/QC methods include:

- Standard Operating Procedures (sample logging, handling and instrument calibration);
- QA/QC Sample Analysis (method blank, matrix spike, duplicate and reference material analyses); and
- Data Reporting Procedures.

2.0 QUALITY ASSURANCE OBJECTIVES

The overall quality assurance (QA) objective is to develop and implement procedures for field sampling, investigation, laboratory analysis and reporting that will provide accurate data of known and documented quality. Data quality objectives (DQOs) for the measurement of data in terms of precision, accuracy, and representativeness are established to ensure that the data collected are sufficient and of adequate quality for their intended use. Data collected and analyzed in conformance with the DQOs process described in these QA/QC procedures will be used to assess the uncertainty associated with pilot study test results.

Evaluation of laboratory data by NTH will incorporate a review of the quality control deliverables outlined in Section 8.0 of this Appendix. Specifically, the quality control objectives will be assessed using the criteria described in the following subsections.

2.1 Precision

Precision is defined as the level of agreement among replicate measurements of the same characteristic. Precision will be evaluated using duplicate analyses. Field duplicate analyses will provide a measure of total system variability. Laboratory duplicates will be an indication of analytical variability. Field duplicates, as well as laboratory duplicates will be collected and analyzed at a minimum frequency of 10% per sample matrix. Thus, both total and component (field vs. lab) variability can be determined. Precision will be expressed either as the relative percent difference (%RPD); relative standard deviation (%RSD); or the "I" statistics (similar to the %RPD). The equations are as follows:

$$\%RPD = 100 \times (X_1 - X_2) / (X_1 + X_2)$$

$$\%RSD = (100/2^{½}) \times [2 (X_1 - X_2) / (X_1 + X_2)]$$

$$I = (X_1 - X_2) / (X_1 + X_2)$$

Where:

X_1 and X_2 are reported concentrations for each duplicate sample and subtracted differences represent absolute values.

A more complete definition of precision, as well as the quantitative limits of these evaluations are included in Section 2.13.

2.2 Accuracy

Accuracy refers to the difference between the measured and the true value of the parameter being measured. Accuracy will be evaluated by analyzing matrix spikes, method standards and quality control samples.

Matrix spikes and standard reference samples will be defined as:

$$\% \text{ Recovery} = \frac{\text{Analytical result for standard or reference}}{\text{True standard value}} \times 100$$

Percent recovery for matrix spike samples will be defined as:

$$\% \text{ Recovery} = \frac{\text{Spiked sample - Unspiked sample}}{\text{True standard value}} \times 100$$

A more complete definition of accuracy, as well as the quantitative limits of these evaluations are discussed in Section 2.13.

2.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represents a characteristic of a population, parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling and analytical programs. The representativeness criterion is best satisfied by logical selection of sampling locations, collection procedures and the analysis of a sufficient number of samples. The sampling procedures presented in Section 4.0 of the main text were developed to insure that samples will accurately reflect the makeup of the matrices of concern.

3.0 SAMPLING PROCEDURES

The following sections describe the sampling procedures that will be followed during this study. Expanded descriptions of the sampling networks and detailed procedures for all phases of sample acquisition, handling and analyses are described in Section 4.3 of the main text.

3.1 Maintenance of Sample Integrity

All sample containers will supplied by the laboratory in accordance with EPA-CLP requirements. Bottle/container types, required volumes, preservatives and holding times are presented in Table 1.

All samples will be cooled in ice while on-site and during shipment. The samples will be packed in durable, sealed coolers and shipped to the contract laboratory at the end of each day of field sampling. A chain-of-custody and laboratory assignment request form will accompany each shipment of samples.

TABLE 1

REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIME

Name	Container	Volume Req.	Preservation	Maximum Holding Time
Total Metals	P,G	200 g	Cool 4° C	6 months

P - Plastic
G - Glass

3.2 Field QA/QC Samples

Duplicate samples will be collected at a minimum rate of one per every ten samples (10%) of each sample matrix. If less than ten samples of a matrix are taken during a particular sampling episode, then one duplicate sample will be collected. A field duplicate sample is collected by immediately splitting the recovered sample into two distinct samples and placing each sample into separate and identical sample containers. Duplicate samples will be handled, transported and analyzed in the same manner as the other collected samples.

3.3 Field Data and Information Acquisition

Documentation of all aspects of sample collection is an integral part of field quality control. Records are maintained as a routine part of quality assurance, including field notebook, chain-of-custody forms/procedures and a field equipment calibration notebook.

Field notebooks are used to record all pertinent information (e.g. odor, visual observations, weather), field measurements, and irregularities or deviations from the prescribed sampling procedure. All entries are dated and initialed for personnel identification.

Dedicated logbooks will be used to record calibration and standardization of field instruments to document this data and determine accuracy and precision parameters, as well as track the calibration, maintenance and repair history. All entries will be dated and initiated for personnel identification.

All notebooks are weather proof, and entries are made with water proof ink. Other aspects of data acquisition and management are discussed in Data Management, Section 7 of the main text.

4.0 SAMPLE CUSTODY

Sample custody procedures to be used during the field investigations and laboratory analysis are based, at a minimum, on EPA guidelines. The following subsections describe the procedures to be used during this pilot study at the test plots in Granite City.

4.1 Field Sample Custody Procedures

Sample custody procedures to be used during the field investigations include: general record keeping, sample documentation, chain-of-custody documentation, documentation of requested analysis, documentation of sample shipment and sample procedures and shipment storage. These procedures are designed and implemented to document sample possession and verify that samples were labeled, documented, handled and tracked correctly, thereby maintaining sample integrity.

4.1.1 Recordkeeping

All documents generated during the project are accounted for through accurate and complete record keeping. These documents include but are not limited to: logbooks, field data records, correspondence, photographic prints and drawings.

All documents, such as field logbooks, field data records and chain-of-custody records, are the responsibility of the Field Supervisor (FS). The FS is responsible for maintaining and distributing the documents to the appropriate personnel. Project logbooks are assigned to key individuals who have specific task responsibilities. All pertinent information will be recorded during the project.

Logbook entries will be dated, legible and contain accurate documentation of on-site project activities. Entries made by individuals will be dated and signed by the individual making the entry.

Field data records will be maintained for each task and the project job number recorded on the initial entry page each day. All in-situ measurements and field observations will be recorded with pertinent information necessary to reconstruct sampling operations. Each day's field data records will be dated and signed by all individuals making entries. The FS and the field team will be responsible for making sure that field data records are completed during field activities and are stored safely to avoid possible tampering. Any lost, damaged or voided records will be reported to the FS. After the field team has completed its work for a particular task, all documents generated during the task will be assembled in the field file. The FS is responsible for assuring that collection, assembly, and inventory of all documents relative to a particular task occurs at the time the project objectives are completed.

When slides or photographs are taken, accurate records of location, date, and the name of the photographer will be maintained.

4.1.2 Sample Documentation

The FS is responsible for adhering to proper custody and documentation practices during all field operations. The primary mechanism for making sure that all the necessary information is recorded for each sample or measurement is the extensive use of field data and sample custody forms. The following forms will be used to identify and record sampling methods, field conditions and other pertinent information. The original records will be maintained on file at the Farmington Hills, Michigan office of NTH Consultants, Ltd.

All sampling procedures, measurements, and observations will be recorded on field sample data sheets and field memoranda will be kept in a field log book. The following information will be documented:

- Sample Type
- Sample Identification No., Sample Location, and Other Pertinent Identifiers
- Date
- Soil Sample Depth Increment
- Sample Equipment: trowel, etc.
- Field Observations (e.g., weather, sample location morphology)
- Appearance of Sample (i.e. color)
- Field Measurements
- Sampler's Identity

All field record entries will be done in ink with no erasures. If mistakes are made, a strike through the incorrect entry will be made. Sample information will be reported on the Sample Data Sheet, Figure 4.

All sample containers will be labeled with the following information:

- Project Number
- Sample Identification Number
- Sample Depth
- Personnel ID
- Time of Sampling

An individual tracking number will be assigned to each sample. For example, tracking number 01-001A-071891-150 will provide the the following information:

Sample Plot - 01

Sample Location - 001

Sample Depth - A (0-3")

Date Sampled - 071891

Personnel ID - 150

Additional information such as the NTH Project Number, sample depth, and time sampled will also be printed on the label. An example of the sample labels to be used are provided as Figure 3, Soil Core/Label Example.

4.1.3 Chain-of-Custody (Figure 5)

Prior to shipping, the samples to the analytical laboratory, a Chain-of-Custody Record will be completed. Separate Chain-of Custody Records will be completed for each sample destination (e.g., laboratory or storage). The request for laboratory analyses is listed on the Chain-of-Custody Record. After transfer of sample possession to subsequent custodians, the Chain-of-Custody Record will be signed by the person taking custody of the sample container.

A duplicate of the Chain-of-Custody Record will be maintained by NTH Consultants, Ltd. The original form will be sent with the sample to the analytical laboratory. After receipt of the samples at the laboratory the condition of the samples will be recorded by the receiver. The Chain-of-Custody procedures will document all sample transactions from collection through analysis. The original copy of the Chain-of-Custody Records will be maintained by the analytical laboratory until NTH Consultants resumes custody.

4.1.4 Sample Tracking Log (Figure 6)

A sample tracking log will be completed at the end of each day. This form tracks the sample through collection, storage, and shipment to the testing laboratories. The sample tracking log along with a copy of the Chain-of-Custody Record and the shipment form will be collected and bound together at the end of each day by the Field Investigator. Each bound package represents all samples shipped on that particular day. These records will then be shipped at the end of the week to the NTH Farmington Hills office. Each days' shipment will then be assigned a shipment number and filed sequentially in the shipment logbook.

As analytical results for the lab are received, copies of the data will be placed behind the appropriate shipment number. This will provide personnel with the ability to quickly reference analytical results. Original analytical laboratory results will be filed in binders by the date received to minimize handling and loss of results.

4.1.5 Sample Storage and Shipment

Samples collected for chemical analysis will be shipped to the analytical laboratory the same day it is collected. All samples will be preserved, shipped and analyzed in accordance with EPA designated holding periods. Table 1 presents a summary of these requirements. All samples will be shipped as environmental samples in accordance with the Department of Transportation (DOT) standard requirements for packages, as specified in 49 CFR 173.24 and 49 CFR 173.6.

Individual sample containers will be packed to prevent breakage and transported in a sealed ice chest or other suitable container.

A label containing the analytical laboratory's name and address will be placed on the container. Glass bottles will be separated in the shipping container by cushioning (i.e., styrofoam or absorbent material) to prevent breakage, and to protect plastic containers from punctures. Cooling media will be placed in the cooler in separate plastic bags and sealed as required. A sealed envelope containing the Chain-of-Custody Record and the Laboratory

Assignment Request Form, Figure 7, will be enclosed in a plastic bag and placed in the cooler or shipping container.

4.2 Laboratory Sample Custody Procedures

This section discusses all of the sample management procedures to be utilized in the laboratory beginning with sample receipt through report generation.

4.2.1 Sample Receipt

When a sample shipment arrives, a laboratory sample custodian will sign and date the chain-of-custody sheet. The custodian will be responsible for inspecting the condition of the sample seal and cross-checking the sample labels with the chain-of-custody record to ensure that there are no discrepancies. Any discrepancies will be resolved before a laboratory control number is assigned to the delivery.

A laboratory control number (consecutive, from a bound logbook) will then be assigned for each delivery. This control number, along with sample identifications, will be clearly marked on all sample container labels.

Appropriate information will then be entered into the logbook and sample control sheet. This information will include sample category code, control number, date received, time, number of samples, person receiving sample, and client code or name. In addition, a priority will be assigned to each sample along with the required completion date for analysis. All sample identifications will be listed and assigned a letter code if needed. Test codes for all requested laboratory tests or groups of tests will also be listed.

Any additional paperwork, including chain-of-custody, purchase order, special instructions, etc. will be stapled to the sample control sheet.

4.2.2 Sample Storage

Samples will initially be transferred only to the designated custodian, or stored in a designated refrigerator within the laboratory, and then handled by the minimum number of people necessary for completion of testing. The custodian will be responsible for transfer of the samples to the analysts for testing; the analysts will then be responsible for continued record keeping, and for keeping a permanent record of the sample description, the tests performed, test results and any abnormalities that may occur during the testing procedures. Unused portions of samples and the corresponding documentation will be returned to the custodian.

A list of completed report control numbers will be provided to the sample custodian daily. The corresponding samples will then be removed to dead storage for a designated period, and be either returned to the client, retained upon special client request, or disposed of properly. Remaining samples from completed analyses will be destroyed or discarded only upon orders from the laboratory director.

4.2.3 Sample Handling

Sample analyses will be scheduled by the supervisor based on required holding times, client requests, and laboratory demands. Each analyst will have responsibility for an assigned list of tests. When an analyst or technician is scheduled to perform a given test, the analyst will enter the identification of the samples to be analyzed on the appropriate analysis work sheet. The analyst will remove the samples from the sample holding area, upon approval of the sample custodian, and note the date and time on the work sheet.

Each analytical work sheet contains specific quality control requirements. Blanks, replicates, spikes, blind spikes, etc. will be assigned by the quality assurance officer. After completion of the analytical run, the analyst will perform any necessary calculations as indicated by the method. The analyst will then date and initial the work sheet and submit the information to the laboratory supervisors. The laboratory supervisors will compare and double-check all data on work sheets, ensuring no transcription or calculation errors. Upon completion of this review, the work sheet will be submitted to quality assurance officer for approval. The QA Officer will review the data, checking calculations and verifying check samples against established quality control criteria for the analytical parameter and sample matrix. The QA Officer will date and initial the work sheet indicating approval.

Finally, all work sheets will be filed by parameter in chronological order. This permits simultaneous reviewing of the results from many samples for a single parameter. These sheets will be retained for at least six years.

5.0 REPORT GENERATION

Completed analytical data will be transferred to a preliminary report. This information will be reviewed by the laboratory manager or his assistants ensuring that there are no transcription or calculating errors. The supervisor will initial the report and relate all unusual results to the designated authority immediately.

control sheets will then be removed from the laboratory and attached to the report package by the person responsible for producing the final report. Additional sample information and special notes will then be added. An invoice number will be assigned, and the report will be generated. Methodology and quality control data (when requested) will be included in the report. The laboratory director will then review and sign the final report.

All raw analytical data and a copy of the laboratory final report will be stored by the analytical laboratory. The laboratory director will ensure that copies of all results will be retained for a least six years, including the permanent binding of work sheets.

6.0 CALIBRATION PROCEDURES

The following calibration procedures will be utilized during field and laboratory operations.

6.1 Field Calibration Procedures

All field measurement instruments will be calibrated, operated and maintained in accordance with the manufacturers instructions and EPA recommended frequency. All instruction manuals will be kept with the corresponding instruments. A summary of these procedures are provided below.

6.1.1 X-ray Fluorescence (XRF)

The XRF will be used for on-site analytical screening tool for lead determinations. The XRF will be calibrated daily for detector response versus the built-in standards and versus six lead site specific soil standards and a reference zinc standard at a rate of every fifth sample of the sample batch or at least one sample per batch. The XRF instrument (Oxford Lab-X 1000) is factory calibrated and manufactured by Oxford Analytical Instruments Limited.

If the XRF can not be calibrated with the six lead site specific standards, six unused lead site specific standards will be used. If the XRF still cannot be calibrated with these standards or if the built-in standards can not be calibrated, then the XRF will not be used that day. The XRF will be sent to the manufacturer for repair.

Routine inspection of all equipment is necessary to identify problems requiring maintenance, so field monitoring is not interrupted and the validity and precision of the data is not adversely affected.

6.2 Laboratory Calibration Procedures

The following sections describe the laboratory calibration procedures that will be followed by the analytical laboratory and the appropriate supplies and equipment needed to perform these calibrations.

6.2.1 Instrumentation

All laboratory equipment will be maintained in a state of complete repair. All instruction manuals will be kept near the corresponding instruments. Maintenance and repair information, service contracts, service records and lists of repairmen available will be included in a bound logbook for each instrument. Attached to each piece of equipment will be a minor troubleshooting guide, and the name or title of the person(s) to be contacted in case of instrument failure.

All instruments will be calibrated according to manufacturers instructions and EPA methodology procedures. Each instrument will be calibrated with a three point standard curve and laboratory blank and will be calibrated at the beginning and the end of the day and at a rate of 10% of the sample batch or at least once per sample batch. Records of calibration, including date, time, standards, standards source and name of operator will be included in the bound logbook.

6.2.2 Instruments, Control Limits, and Corrective Actions

The following instruments and instrument control limits for this project are as follows:

- Atomic Absorption Spectrophotometer Flame Technique (control limits ± 2.5 nm wavelength accuracy)

All instruments will be calibrated according to manufacturer's recommendations. Instruments that can not be calibrated accurately (e.g. exceeds instrument control limits) and are verified by the Laboratory Manager will not be used for analysis until the instrument is repaired by the manufacturer.

6.2.3 Standard Calibration and Traceability

All standards and reagents will be purchased and prepared in accordance with EPA-approved procedures, and will be of analytical reagent grade quality or better. Proper selection, preparation, labeling and storage of both reagents and standards will be followed for the particular analytical method. Instructions for specific reagents and standards preparation will be included in the written analytical procedures.

All purchased reagents will be labeled with date of receipt and stored in separate supply rooms, away from traffic and laboratory hazards.

All prepared reagents and standards will be labeled with the preparation date, expiration date, concentration, and name of the preparer. Prepared standards and reagents will be stored as close to relevant analytical work areas as possible to maximize work efficiency. All reagents and standards will be checked for expiration date at the beginning of each work day. Expired reagents and standards will be discarded and freshly prepared standards and reagents will be purchased and/or prepared. Pre-prepared standards will be ordered from either the National Bureau of Standards, or alternative standards traceable to NBS or the USEPA.

6.2.4 Routine Laboratory Operations, Instrument Logs, Supplies

For inventory maintenance, all supplies, including reagents, standards, glassware and replacement parts will be ordered through or by the laboratory supply clerk. Current supplies will never be allowed to fall below a two-month requirement. When removing supplies, the current inventory and established minimum supply requirements will be considered, at which time re-orders may be necessary. Order forms will be used, including all pertinent information, such as quality, quantity, mesh size or other physical or chemical properties.

An inventory will be taken once a month and expired reagents and standards will be discarded. All balances will be listed in their respective logbooks by manufacturer, model, and serial number, noting applicable repair information. The balances will be kept on shock-resistant tables away from laboratory traffic. The area around the balances will be kept clean and free from other equipment. The balances will be checked weekly with NBS-certified weights. Annual service visits will be made by the manufacturer under a service contract. Dates of all calibration adjustments will be noted in the maintenance logbook and initialed by the personnel performing the calibration.

In general, all instruments will be listed by manufacturer, model, and serial number in a logbook, including maintenance, repair, service, and replacement parts information. All instruments will be calibrated according to manufacturer's recommendations.

6.2.5 Ovens, Incubators, Refrigerators, etc.

Thermometers (of the appropriate temperature range and graduations) will be placed inside equipment to ensure correct temperature. If necessary, more than one thermometer will be placed in a sand bath or in viscous liquid (e.g. glycerol) to prevent rapid change in the reading while gaining access to the equipment interior. A daily log will be maintained on each instrument by recording temperature, time and technician's name. The equipment will be calibrated if necessary and recorded on the posted log sheet. When sheets are completed, they will be placed in the equipment logbook.

Thermometers will be standardized yearly against NBS or an equivalent certified thermometer. They will be tagged with date and correction factor, which will be recorded in the maintenance log. Correction factors will always be applied.

6.2.6 Compressed Air, Electricity, Hoods, and Tap Water

Any conductance of malfunction or interruption of these laboratory services will be immediately reported to the laboratory supervisor, the laboratory manager, or the laboratory director.

6.2.7 Laboratory Pure Water

The conductance of deionized water will be monitored daily and recorded on a posted laboratory distilled water form. The conductivity must be less than 1 μmho/cm. All completed water quality forms will be stored in the logbook. The laboratory manager will be notified immediately if the water is unsatisfactory so that prompt action may be taken. The recharging of the deionizer or any change in distillation apparatus will be recorded.

A complete check for contamination will be made whenever appropriate.

6.2.8 Glassware

Borosilicate glassware will be used based on laboratory requirements, with the use of Kimax or Pyrex brands for most analyses. Any glassware that is chipped, cracked, or shows signs of excessive etching will be discarded. Only class "A" volumetric ware will be used. Disposable glassware will be used only when economically feasible, and only if it's use has been established to have no effect on quality assurance. Disposable glassware can be very useful in cases where cross-contamination might occur.

Chemically inert plastic ware, when appropriate to specific methodology, may be ordered and used.

Cleaning methods will be selected to match the type of contamination being removed. Acid solution detergents or organic solvents will be used as required to remove oil, grease, and other contaminants. Persistent contamination may require nitric acid, chromic acid or aqua regia. For final washing, tap-water will be used followed by at least three successive rinses with distilled water.

7.0 **ANALYTICAL PROCEDURES**

All sample analyses will be performed in accordance with published methodologies. These methodologies include the following references:

USEPA, **"Methods for Chemical Analysis of Water and Wastes"**, EPA-600/4-79-020, Revised March 1983.

USEPA, **"Test Methods of Evaluating Solid Waste"**, SW-846, Third Edition, November 1986.

ASTM, **"Annual Book of ASTM Standards"**, Volume 04.08, 1990.

Method procedural manuals will be updated to accommodate reference method revisions, new instrumentation, instrument peculiarities, etc. Suggestions from the supervisors, analysts and technicians will be received by the laboratory manager. Any changes must be approved and reviewed by the Laboratory Director prior to insertion in the procedural manual.

Each analytical parameter has a primary methodology selected according to sample matrix, individual contaminant levels, the number of contaminants and other possible interferences present. Secondary methodologies will be performed only upon approval of the laboratory manager.

Routine sample preparation procedures will be included in the procedural manual, appearing with the appropriate analytical method. Specifically, the following analytical methodologies will be used for the sample mediums specified:

1. **Analysis of soil samples will be analyzed using USEPA, "Test Methods for Evaluating Solid Waste", SW-846, Third Edition, November 1986 for inorganic analyses. Specific methods that will be used for the chemical analysis samples collected during this study are shown in Table 2 of this section.**

2. Physical testing of field soil samples will be performed in accordance with methodologies described in the ASTM, "Annual Book of ASTM Standards", Volume 04.08, 1990. Specific methods that will be used for the physical soil testing during this study are shown in Table 3 of this section.

TABLE 2

Summary of Analytical Program for Soil

PARAMETER	TOTAL # OF SAMPLES	EPA (1) METHOD	REPORTING UNITS
Lead (1)(2)	200±	7420	mg/kg

(1) USEPA, "Test Methods of Evaluating Solid Waste", SW-846, Third Edition, November 1986.

(2) Samples are screened in the field by X-Ray Fluorescence XRF), see Section 4.4 in the main text for methodology.

Analytical data will be validated following the guidelines presented in USEPA's Laboratory Data Validation, Functional Guidelines for Evaluating Inorganic (June 13, 1988) Analyses.

TABLE 3
Summary of ASTM Physical Analytical Program for Soil Samples

TEST METHOD CONTENT	METHOD NO.
Particle-Size Analysis	D422
Liquid Limit, Plastic Limit, and Plasticity	D4318
pH of Soils	D4972
Moisture Content	D4643

All of the methods referenced in Table 3 are taken from "Soil and Rock; Dimension Stone; Geosynthetics", Volume 04.08, Annual Book of ASTM Standards, 1990

Standard operating procedures outlined in the above referenced methods will be followed for all analytical parameters. The methods selected above were based on the type of sample.

The detection limit is defined as the lowest quantifiable detectable level above instrument sensitivity.

8.0 DATA REDUCTION, VALIDATION AND REPORTING

To ensure that proper data reduction, reporting and validation procedures are employed, project QA officials will review the field and laboratory data. Evaluation of the data will incorporate a review of the Quality Control Objectives (QCO) in terms of precision, accuracy and representativeness.

8.1 Precision

Precision will be evaluated using duplicate analyses. Field duplicate analyses will provide a measure of total system variability and laboratory duplicates will be an indication of analytical variability.

8.2 Accuracy

Accuracy refers to the difference between the measured and the true value of the parameter being measured. Accuracy for laboratory analyses will be evaluated by analyzing matrix spikes, method standards and laboratory quality control samples. Accuracy for field pH and specific conductance will be determined by analyzing quality control samples.

8.3 Representativeness

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variation at a sampling point, or an environmental condition. Representativeness is a qualitative parameter which is most concerned with the proper design of the sampling program. The representativeness criterion is best satisfied by logical selection of sampling locations and collection of a sufficient number of samples. The sampling procedures presented in Section 4.0 of the main text were developed to insure that samples will accurately reflect the makeup of the matrices of concern.

8.4 Data Validation

Data will be validated following the guidelines presented in USEPA's Laboratory Data Validation, Functional Guidelines for Evaluating Inorganic (June 13, 1988) Analyses.

Once the entire package has been reviewed, a narrative report and quality assurance objective summary will be prepared. This report will indicate the quality of the data and identify any specific problem areas.

For each parameter analyzed, the following is a list of the appropriate quality assurance tasks that will be followed during this Pilot Study.

Metals

- Chain of Custody (field and laboratory)
- Holding Times
- Calibration Data (Initial and Continuing)
- Blanks
- Laboratory Control Sample
- Laboratory Duplicate Samples
- Matrix Spike Sample
- Atomic Absorption Direct Aspiration QC
- Sample Result Verification
- Field Duplicate

8.5 Reporting

The laboratory will provide a written Certificate of Analysis for each batch of samples. Each page of the report and associated backup material will be marked with the delivery order number and the laboratory's name and address. The report will contain the date of collection and results of each analysis printed on the laboratory's letterhead form, and will be certified by the laboratory director. Backup materials attached to each report will include copies of relevant notebook pages, standard calibration curves, and output from automated data collected systems (both graphic and numeric displays). A Quality Control Report will be included showing method blanks, standards, precision and accuracy data derived from the quality control samples.

9.0 QUALITY CONTROL CHECKS

The following Quality Control Checks will be used during field and laboratory operations. A summary of this information is presented in Table 4 of this section.

9.1 Field Quality Assurance/Quality Control Procedures

This section describes the field QA/QC sample collection procedures for field duplicate samples.

TABLE 4

QUALITY ASSURANCE
PROJECT OBJECTIVES SUMMARY

QUALITY OBJECTIVES	FREQUENCY
Chain-of-custody	Every sample
Holding times	As shown on Table 1
Field duplicates	10% of similar medium- Minimum one per sample medium
Analytical blank	10% - One minimum
Matrix spike (recovery)	10% - Minimum one per analytical run
Laboratory duplicates	10% - Minimum one per analytical run
Laboratory replicates	10% - Minimum one per analytical run
QA reference sample	10% - Minimum one per analytical run
Blind check samples	As necessary
Calibration data	Initial and as specified by EPA for specific methodology
Detection limits	As specified by EPA for specific methodology
Method detection limits	As required
Concentration and instrument response	To determine calibration/ standard curve linearity and validity of data

9.1.1 Field Duplicate Samples

Field duplicate samples will be collected to evaluate sampling reproducibility. Duplicate samples will be collected at a minimum rate of one per every ten samples (10%). If less than ten samples of a matrix are taken during a particular sampling episode, then one duplicate sample will be collected.

- Duplicate samples will be collected by splitting the recovered sample, then placing one part in the regular sample container and the other portion in the duplicate sample container.

9.1.2 Field Data Proofing

Data transcription and data cross-calculation errors are checked for by the On-Site Field Supervisor in all of the daily field reports.

9.2 Laboratory Quality Assurance/Quality Control Programs

This subsection describes the laboratory QA/QC procedures to be utilized.

9.2.1 Control Samples

Control samples are those samples that are introduced into the train of actual samples as a monitor on the performance of the analytical system. A control sample may consist of standard or natural matrices. Analysts will be responsible for performing control samples at a rate of 10% of the sample batch or at least one per sample batch, as they are required in the methodology work sheets. Some analytical parameters will require only duplicates, while others may require duplicates, midpoint standards, matrix spikes and blanks.

9.2.2 Types of Control Samples

Duplicates, Midpoint Standards and Control Samples

Provide indications of the precision of the analytical system. They will not provide indications of matrix effects or accuracy.

Blanks

Provide an indication of positive interferences introduced within the laboratory. They will not provide information on matrix effects, accuracy, precision or natural background.

Matrix Spikes

Can provide information on matrix effects, precision, natural background and accuracy.

9.2.3 Introduction of Control Samples

With every batch of samples performed for the same parameter, the analyst must perform blanks, midpoint standards, duplicates and matrix spikes according to the work sheet specific to that parameter and sample matrix.

In general, a blank, midpoint standard, duplicate and matrix spike will be performed with every ten samples. These quality control samples will be introduced by the analyst. The spike solution and designated amount to be introduced will be presented to the analyst upon his request by the QA Officer. When deemed necessary by the QA Officer, blind check standards (which can be blanks, duplicates, standards, or matrix spike samples) will be introduced into the laboratory as samples, complete with control numbers and promised dates.

Preparation of Laboratory Duplicate Samples

A laboratory duplicate sample will be obtained by transferring two separate sample volumes, then preparing and analyzing them separately. In general, duplicate samples will be performed every 10 samples.

Preparation of Laboratory Matrix Spike Samples

Matrix spike samples will be obtained by transferring a known sample volume, adding a known amount of standard, then preparing and analyzing the sample.

Laboratory Standard Reference Samples

All standards will be purchased and prepared in accordance with EPA protocol.

9.3 Reference Materials

Samples of standards for chemical analyses will be based on reference materials which are either National Bureau of Standards (NBS), Standard Reference Materials, or can be traceable to one of these materials. NBS Standard Reference Materials will be accepted at face value; however, care will be taken to be sure that the standard will be used for the purpose for which it was intended.

Although NBS offers only a limited selection of standards, it is possible to trace any substance through a series of comparisons to some other standard reference material.

10.0 PROCEDURES FOR THE DETERMINATION OF DETECTION AND QUANTIFICATION LIMITS

The method detection limit will be defined as the minimum concentration of a substance that can be identified, measured and reported with 99 percent confidence that the analyte concentration is greater than zero, and determined from analysis of a sample in a given matrix containing analyte. The methodology, referenced in Table 2 has an established detection limit for the parameter to be analyzed. This method detection limit will be used for all sample analysis.

Quantification Limits may vary with sample matrix, therefore every effort to complete all quality control procedures will be made prior to data reporting.

In general, reporting limits will be established for each parameter and matrix type. If an analyte is quantifiable below an established reporting limit, this value will be reported. However, the analyst must perform the complete procedure for the determination of the method detection limit.

10.1 Laboratory Data Proofing

Data transcription and data cross-calculation errors are checked by the Laboratory QA Officer in all of the daily analytical data. Unacceptable data will be considered invalid and slash will be drawn through the unacceptable data and explanation of why the data has been considered unacceptable will be entered next to the invalid data. The page(s) will be reviewed and signed by the laboratory analyst and laboratory QA Officer.

11.0 PERFORMANCE AUDITS

11.1 Field Operation Audit Procedures

For this pilot study it is not necessary to perform audits, however field operation review audits may be conducted during the course of the study if some inconsistencies or problems become evident. This field audit review will verify that the field methods and sampling procedures detailed in the Statement of Work are followed. The audit will be performed by the NTH QA Officer or a designated person, chosen by the NTH QA Officer independent of the sampling team to maximize the objectiveness of the audit. The person(s) performing the audit will be familiar with the Statement of Work and will have had previous experience with the procedures that are subjected to the audit. The field procedures that will be audited include sampling techniques. The audits will be conducted using a Field Surveillance Checklist developed from the procedures for each task. Any deficiencies or discrepancies from the QA/QC plan revealed during the audit will be discussed and corrected with the On-Site Field Supervisor. An audit report will be distributed to project managers and QA officials. The field audit procedures and findings will be documented and discussed in the final report.

11.2 Laboratory Performance Samples

The Laboratory QA Officer is responsible for maintaining the continuity of the laboratory's ongoing inter-laboratory analysis program by ensuring that:

- Performance samples are received from the various sources, and analyzed and reported in a timely fashion.

- A sufficient number of performance and reference samples are acquired to include as many of the relevant analytical parameters as are available.

- The results received from the various agencies are compiled and placed in the computer audit section, and reported to the laboratory management.

11.3 Laboratory Audit Procedures

Laboratory review audits may be conducted by the NTH QA Officer or by a designated person, chosen by the NTH QA Officer if any inconsistencies in the results from laboratory analysis is noticed. If audit procedures are deemed necessary, it will include a review of sample custody and handling procedure, analytical methods, QA/QC objectives, data management and reporting procedures. A Laboratory Audit Checklist Form will be developed from the protocol outlined in this QA Project Plan will be utilized during audit activities. Any deficiencies or discrepancies from the QA/QC plan discovered in the audits will be discussed and corrected with the Laboratory QA Officer. Audit reports will be distributed to project managers and QA officials. Audit procedures and findings will be documented and discussed in the final report.

12.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION AND ACCURACY

12.1 Precision

Precision measures the reproducibility of measurements under a given set of conditions. Specifically, it is a quantitative measure of the variability of a group of measurements compared to their average value.

Precision is usually stated in terms of standard deviation but other estimates such as the coefficient of variation (relative standard deviation), range (maximum value minus minimum value), and relative range are common, and may be used pending review of the data.

The overall precision of measurement data is a mixture of sampling and analytical factors. Analytical precision is much easier to control and quantify than sampling precision. In contrast, sampling precision is unique to each site.

Sampling precision will be determined by collection of field duplicate samples. Analytical results from laboratory duplicate samples will provide data on overall measurement precision.

Subtracting the analytical precision from the measurement precision will define the sampling precision. Precision will be expressed as with the relative percent difference (%RPD); relative standard deviation (%RSD); or the "I" Statistic:

$$\%RPD = 100 \times (X_1 - X_2) / (X_1 + X_2)$$

$$\%RSD = (100/2^{1/2}) \times [2 (X_1 - X_2) / (X_1 + X_2)]$$

$$I = (X_1 - X_2) / (X_1 + X_2)$$

X_1 and X_2 are reported concentrations for each duplicate sample and subtracted differences represent absolute values. The equations are taken from Data Quality Objectives for Remedial Response Activities (EPA/540/G-87/003; March 1987).

Field and laboratory duplicates will be collected and analyzed at a frequency of a minimum of one duplicate per 10 samples (10%) per given matrix. If less than 10 samples per day of a particular medium is collected or analyzed, then a minimum of one duplicate sample will be obtained.

Initially, precision data will be assessed using predetermined limits. The initial predetermined limits are dependent on the type of analysis and have selected in accordance with EPA approved procedures referenced in 40 CFR 136 and SW-846. They are as follows:

- Metals: < 20% RPD or equivalent

Control charts will be used to monitor the day-to-day variations in the precision, and to detect trends in these variations. The construction of control charts requires an initial database to establish the mean and standard deviation of measurements so that working warning limits and control limits can be established. However, at no time will data acceptance criteria become less stringent than the initial control limits specified above. A more detailed discussion on how control charts will be established is included in Section 2.14.

12.2 Accuracy

Accuracy measures the bias in a measurement system. Since it is difficult to measure for the entire data collection activity, accuracy will be assessed through use of known QC samples and matrix spikes.

Accuracy values can be presented in a variety of ways. The average error is one way of presenting this information; however, more commonly, accuracy is presented as percent bias or percent recovery. Percent bias is a standardized average error; that is, the average error divided by the actual or spiked concentration and converted to a percentage. Percent bias is unitless and allows accuracy of analytical procedures to be compared easily. Percent recovery provides the same information as percent bias.

Percent recovery for internal QC samples and standard reference samples will be defined as:

$$\% \text{ Recovery} = \frac{\text{Analytical result for standard or reference}}{\text{True standard value}} \times 100$$

Percent recovery for spike samples will be defined as:

$$\% \text{ Recovery} = \frac{\text{Spiked sample - Unspiked sample}}{\text{True standard value}} \times 100$$

These equations are taken from <u>Data Quality Objectives for Remedial Response Activities</u> (EPA/540/G-87/003; March 1987).

Initially, accuracy data will be assessed using parameter specified predetermined limits. The initial predetermined limits have been selected in accordance with EPA approved procedures referenced in 40 CFR 136 and SW-846. They are as follows:

- Metals: $\pm$ 20%

Control charts will be used to monitor the day-to-day variations in the precision, and to detect trends in these variations. The construction of control charts requires an initial database to establish the mean and standard deviation of measurements so that working warning limits and control limits can be established. However, at no time will data acceptance criteria become less stringent than the initial control limits specified above. A more detailed discussion on how control charts will be established is included in the following section, 13.

13.0 QUALITY CONTROL CHARTS

Control charts will be used to monitor the day-to-day variations in the precision or accuracy of routine analyses, and to detect trends in these variations. However, at no time will the acceptance criteria be less stringent than the initial criteria. The construction of control charts requires an initial database to establish the mean and standard deviation of measurements. The database consists of measurements obtained from performing the complete analytical method.

The mean percent recovery will be the central line of the control chart. The upper warning limit (UWL) and lower warning limit (LWL) will be established at one times the standard deviation above and below the central line, respectively. The UWL and LWL are calculated as follows:

$$UWL = Mean + (1.000 \times Standard\ Deviation)$$

$$LWL = Mean - (1.000 \times Standard\ Deviation)$$

The upper control limit (UCL) and lower control limit (LCL) will be established at two times the standard deviation above and below the central line, respectively. The UCL and LCL are calculated as follows:

$$UCL = Mean + (2.000 \times Standard\ Deviation)$$

$$LCL = Mean - (2.000 \times Standard\ Deviation)$$

The purpose of determining, using, and enforcing control limits is to guarantee an element of precision (meaning predictable reproducibility) in a procedure. The warning limits are based on a 68.0% confidence level, where the data that exceeds this confidence level but below the upper control limit are valid. The control limits are based on a 95.0% confidence level. There is always a small probability that 5.0% of the data will fall outside the control limits.

13.1 Control Chart Validation

Historical laboratory data results will be used to determine control limits and new data will be plotted sequentially against these limits. The results will be evaluated as follows:

- check values within control limits but outside warning limits give early warning of problems.

- check for values outside control limits show definite problem area to be corrected.

- Runs of six or more may indicate a bias in the results even if the results are within control limits. (A run is a sequence of results all above or below the average of the process.)

Values above the warning limits but below the control limits will be checked for Determinate errors: instrument error (calibration, reagent purity,...), operative errors (blunders, carelessness, mistakes), personal errors (handicaps, prejudice), errors of method (inherent in the method, independent of operator and equipment, i.e. - side or incomplete reactions,....); and random errors: errors brought about by the effects of uncontrollable variables, or due to the limits of precision of the instrument or operator.

Errors found as a result of values exceeding the warning limit but below the control limit will be corrected before any further analysis is performed.

Six or more consecutive points on the same side of the average percent recovery, as well as values beyond the control limits, will be judged out-of-control. The determinate and/or random errors will investigated and corrections will be implemented before any further analysis is performed.

14.0 CORRECTIVE ACTION

When a problem situation arises regarding any significant impediment to the progress of the investigation as detailed in the Statement of Work, corrective action will be implemented to identify the problem and its source. Appropriate documentation of this action will be recorded in the project file.

The predetermined limits for data acceptability are specified in this section. Corrective action will also be taken when data are determined to be suspect due to lack of correlation with past monitoring events.

When, for any reason, there is an out-of-control situation, the out-of-control sample(s) and all previous samples analyzed from the last in-control sample through to the out-of-control sample(s) will be rerun.

No data will be discarded unless sufficient reason can be cited to justify it (i.e. - a point beyond control limits is not sufficient justification to discard the point.)

When sample(s) that are rerun remain out-of-control, efforts will be made to determine the cause, and corrective actions will be taken to bring the process under control. The analyst or analysts involved, supervisors and laboratory management will be consulted. The out-of-control situation and corrective actions taken will be fully documented. Data obtained since the last in-control QC sample will be considered invalid. Analysis of samples will not continue until the process is determined to be in control.

Personnel responsible for the initiation of and approval of corrective action will be the QA officers assigned to the project.

APPENDIX C

QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR PUBLIC HEALTH AND SAFETY MEASURES

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APPENDIX C

QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR PUBLIC HEALTH AND SAFETY MEASURES

1.0 INTRODUCTION

This appendix contains the Quality Assurance/Quality Control (QA/QC) procedures for the Public Health and Safety Measures presented in Appendix A. Air Monitoring presented in Appendix A follows EPA air sampling procedures contained in 40 CFR Part 50 Appendix B, Reference Method for Determination of Suspended Particulate Matter in Atmosphere (High Volume Method), and the analytical procedures contained in 40 CFR Part 50 Appendix G, Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air. For comparability to EPA air quality standards, it is critical that QA/QC procedures be followed.

2.0 QUALITY ASSURANCE (QA) OBJECTIVES

The precision goal for all analyses will be less than 15%. The accuracy goal is 85% to 115% and the completeness goal is 95%. Procedures for calculating precision, accuracy and completeness are presented in Section 10.0 of this document.

APPENDIX C

QUALITY ASSURANCE/QUALITY CONTROL PLAN FOR PUBLIC HEALTH AND SAFETY MEASURES

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3.0 SAMPLE CUSTODY

Each and every sample filter will be labeled with a serial number. The transport envelope will be labeled with the following information: project number, start date and time, finish date and time, sampler's name, serial number of filter, and Hi-vol unit number. A chain of custody form will be completed for each sample and will remain with each sample throughout the project. A sample tracking log will be maintained of all samples including the date when they were transferred to the analyst from the sampling person.

4.0 ANALYTICAL PROCEDURES

Sample analysis will be performed in accordance with 40 CFR 50, Appendix G, Reference Method for the Determination of Lead in Suspended Particulate Matter Collected from Ambient Air.

5.0 CALIBRATION PROCEDURES AND FREQUENCY

The sampling equipment will be calibrated at the beginning of the project and once during the project. Hi-Vol samplers will be calibrated using a five point orifice plate set and manometer.

6.0 DATA REDUCTION, VALIDATION AND REPORTING

At standard temperature and pressure, sample air volume will be calculated as follows:

$$V = QT$$

Where:

V = Air volume sampled (m^3)

Q = Average sampling rate (m^3/min)

T = Sampling duration (minutes)

For each sample, the lead concentration will be calculated as follows:

$$C = \frac{(L \times 100 \times 12) - B}{V}$$

Where:

C = Lead concentration (ug/m^3)

B = Blank lead value (ug)

L = Lead content of filter strip (ug)

V = Sample volume (m^3)

For each sample, total suspended particulate mater concentration is calculated as follows:

$$TSP = \frac{(W_f - W_i) \times 10^6}{V}$$

Where:

TSP = Mass Concentration of Total suspended particulate matter (ug/m^3)

W_i = Initial weight of clean filter, g

W_f = Final weight of exposed filter, g

V = Air volume sampled, converted to standard conditions, m^3

Data validation is an on-going procedure throughout the program. The principal criteria used to validate the integrity of the data include the following:

- **Frequent verification that all raw data generated in the preceding week have been stored on magnetic tape and/or in hard copy, and that storage locations have been documented in the laboratory chain-of-custody records;**

- **Examination of at least five percent of the raw data on a frequent basis to verify adequacy of documentation, confirm peak shape and resolution, assure that the automatic integrator was sensing peaks appropriately, and so forth;**

- Reporting of all associated blank, standard, and QC data along with results for analyses of each batch of samples; and

- Reporting of all analytical data for samples with no values rejected as outliers, because of the small number of replicate samples for analysis.

7.0 INTERNAL QUALITY CONTROL (QC) CHECKS

Internal quality control checks shall fall into three basic areas. Each of these areas is presented below.

Blanks

Blank samples are analyzed to assess possible contamination from the field and or laboratory so that corrective action can be taken if necessary. Blank samples include the following:

- Field Blanks - These are exposed to field sampling conditions. One field bank will be collected for each day of field testing.

- Method Blanks - One set of blank samples is prepared in the laboratory for each lot of samples to assess possible laboratory contamination.

- Reagent and Solvent Blanks - One set of blanks is prepared for each new lot number of solvent or reagent to determine the possible background of these materials.

Analytical Replicates

Replicate analysis will be performed on approximately 10% of all samples.

8.0 PERFORMANCE AND SYSTEM AUDITS

System audits shall include frequent and ongoing reviews of data by the quality assurance manager to see that all QC procedures are carried out and that the results are satisfactory. Blind audit samples may be generated and submitted to the laboratory for analysis as a means of checking performance quality. The results of the analysis of these samples will also be used for the purpose of calculating analytical accuracy.

9.0 PREVENTATIVE MAINTENANCE

Preventative maintenance checks and repairs of all sampling and analytical equipment will be incorporated into all routine calibration checks.

10.0 SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA PRECISION AND ACCURACY

Precision is measured in terms of the relative standard deviation (RSD) of the analysis of replicate samples. This is calculated as shown below.

$$RSD\ (\%) = \frac{SD}{t} \times 10$$

Where:

SD = Standard deviation

t = Average of sample set

$$SD = \frac{\sum_{i=1}^{n} (C_i - C)^2}{n - 1}$$

C = Mean concentration of the set of replicates

C_i = Concentration in the sample

n = Number of replicates

Accuracy will be evaluated by comparing the analytical results for blind audit samples to the actual values for these samples. Accuracy will be calculated as follows:

$$A = \frac{M_r}{M_a} \times 100$$

Where:

A = Accuracy (%)

M_r = Mass reported by the laboratory (ug)

M_a = Actual mass (ug)

11.0 CORRECTIVE ACTION

The need for corrective action is evaluated on the basis of the ongoing analysis of precision and accuracy. When the goals specified in Section 2.0 of this appendix are attained, no action will be taken. If, however, the performance drops below the specified goals, corrective action will be taken immediately. The basic process involved in taking corrective action involves the following seven steps.

1. The problem is defined.

2. Responsibility for investigating the problem is assigned

3. The cause of the problem is investigated and determined.

4. A corrective action to eliminate the problem is determined.

5. Responsibility for implementing the corrective action is assigned and accepted.

6. The effectiveness of the corrective action in eliminating the problem is verified.

The specific action to be taken is dependent upon what is determined to be the cause of the problem.

12.0 QUALITY ASSURANCE REPORTING

The project staff will discuss progress and plans weekly. A QA/QC report will be generated periodically. This report will be discussed by the project team and plans will be made to correct any areas of weakness.

in all cases.

b. Call the retainer by name.

c. If the refusal is anything other than bedrock, give an explanation as to what type of refusal occurred. (i.e. boulder, caliche zone, etc...)

49. Page 2-5, Section 2.3.2 (SOP 1)

Samples will be "homogenized", not composited.

50. Page 2-7, Section 2.6.1 (SOP 1)

Field logbooks shall not include project costs. Only pertinent field information should be included in the logbooks. Costs are a program issue to be addressed elsewhere.

51. Page 2-8, Section 2.6.2 (SOP 1)

a. Use the boring log supplied in the Scope of Services.

b. Supply the driller's name.

c. Use a 1' = 1' scale.

d. Include the plasticity, grain size, and percent of materials, in addition to the items mentioned.

52. Page 2-2, Section 2.2.1 (SOP 2)

It is not necessary to explain the drilling technique, but be more specific on the type of rig, type and size of augers, procedures to be followed, etc...

53. Page 2-2, Section 2.2.2 (SOP 2)

a. Add bottom, vented cap, and development forms to the list.

b. Logs are already explained in SOP 1. Reference to stratigraphic logs makes it sound like this is another type of log to fill out.

c. It appears to be redundant to also write the log in the field logbook. Is the field logbook a stand alone document with no supporting data?

d. Logging will be done on the stainless steel split spoon soil samples.

e. Sample cuttings will not be used for logging unless spilt-spoon sample recovery is not sufficient to allow for logging by this method.

f. Should read -Surface elevation ("when" available).

g. Add sample ID and type of sample after the sample depths and times.

h. Water levels should be both the first encountered water level and the 24-hour reading.

i. Geotechnical samples will be collected in glass/plastic jars, and sealed with three (3) wraps of electrical tape (not plastic bags).

j. Samples taken for moisture content should be taken in moisture tins and sealed.

54. Page 2-3, Section 2.2.3 (SOP 2)

Joints will not be glued and lead shot will not be used. Caps will be vented.

55. Page 2.4, Section 2.2.3 (SOP 2)

a. Continuous wrap, not factory slotted well screens will be used according to the Scope od Services. the well screens will have an inside diameter "equal to" the well casing (not greater than).

b. Soil samples from the screen interval will be tested to verify the screen slot size choice. How was the slot size chosen?

c. How was the filter pack chosen?

d. A buffer sand is not needed above the seal if the seal is hydrated properly with a buffer sand below the seal.

56. Page 2-5, Section 2.2.3 (SOP 2)

a. A bag of cement is 96 pounds. How much water will be added to the mix?

b. Do the state regulation allow for a 10% bentonite mix?

c. The tremie pipe will be constructed such that the grout will be directed horizontally and not vertically.

d. ID tags will be attached to the protective casing for easy reference.

e. Guard posts will be filled with cement if they are hollow.

57. Page 2-6, Section 2.2.4 (SOP 2)

Will the wells be installed inside the augers? If so, clarify. Will centralizers be used if constructed by this method?

58. Page 2-8, Section 2.3 (SOP 2)

Add the information requested in the Scope of Services for the ID tag.

60. Page 3-1, Section 3.1 (SOP 2)

a. Add turbidity (nephelometric) meter to the list.

b. How will the turbidity meter and temperature meter be calibrated.

61. Page 3-3, Section 3.2 (SOP 2)

a. Development should consist of surging and pumping "up to" 4 hours.

b. Air lifting will continue "up to" 4 hours. If parameters have not stabilized in that time frame, an additional four hours may be needed. When at least five volumes have been removed and the parameters have stabilized, development is complete. Anything over 8 hours for development would need approval by the USACE project manager.

d. When will sediment accumulation be measured? Should be very close to sediment free (6-inches is too much).

e. Each well volume or 10 bailers full, which ever comes first.

62. Figure 1 (SOP 2)

a. The buffer sand is not shown on the detail.

b. Show the distance that the screen is off the bottom of the hole.

c. The pad should be 4" not 4'.

63. Page 2-2, Section 2.1 (SOP 3)

a. Add chain of custody seals to the list.

b. Add temperature meter and calibration procedures.

64. Page 2-4, Section 2.1 (SOP 5)

a. What about chain of custody seals?

b. What about cooler preparation for shipment? Be specific.

65. Page 2-2, Section 2.2 (SOP 8)

Why total depth?

6.2 Field Operation

The field work during this pilot study will involve two distinct phases; homogenization of the near-surface soils and application of lime and nutrients. The following sections discuss both operations.

6.2.1 Homogenization

The primary focus of the field operation is to determine the efficiency and effectiveness of soil homogenization. Since the test plots will be selected based upon lead in the surface soils, efficiency will be evaluated based upon the final distribution of lead after homogenization of the soils to a depth of nine inches. The number of passes and type of tilling/mixing equipment will be two physical parameters studied while the variability of the lead content within the test plots will be the chemical parameter monitored. Each test plot will be homogenized vertically from 0-9". Homogenization of soil will continue, in the form of equipment passes, until field tests indicate that the lead distribution has approached a constant. The pass number, samples obtained, and comments will be recorded on the Field Sampling Form, Figure 10.

Four of the selected plots will be tested to determine the optimum tilling/mixing effort for homogenization. Initially, two to three passes may be required to meet the required tilling depth of nine inches. Adjustments will be made, as required, during the field operation.

The degree of homogenization will be analyzed in the field by computing the mean and standard deviation of lead concentrations using XRF test results from samples collected after each pass. Homogenization will continue until the mean and standard deviations from consecutive passes approach a constant. A data curve similar to Figure 11, Plot of Mean Soil Lead Concentration Versus Number of Passes, will be used to determine the optimal number of equipment passes required to achieve relatively uniform mixing.

Once the optimal number of passes has been determined for homogenization, 24 samples will be collected and placed in 125 ml sample jars and shipped to Martel Laboratory Services, Inc. for analysis. Each jar will be clearly marked with project name and number, test plot, sample depth, date, time, and name of the field technician collecting the sample. Standard chain-of-custody procedures will be followed, as discussed in Section 4.0. The total lead content (EPA Method 7420) of each sample will be determined to assure accurate correlation with XRF results and to provide a more precise determination of lead concentrations and distribution throughout the test plot.

During the homogenization operation, air sampling will be performed to assure that ambient air quality standards are not exceeded. Personal air monitoring will also be used by personnel directly involved in the homogenization operations.

6.2.2 Soil Treatment Using Lime and Nutrients

Lime will be applied to eight (8) test plots after each plot has been homogenized. Nutrients will be applied to all sixteen (16) test plots. The quantities of lime and nutrients to be added will be based on recommendations provided by the Soil Science Department of Michigan State University, as detailed in Section 5.3.1. Lime purity and fineness will be considered according to standard agricultural practice. This will assure maximum effectiveness of the applied lime in terms of neutralizing soil acidity and reaction with lead compounds. Nutrient additions, such as nitrogen, phosphorous, potassium, magnesium and calcium, will also meet MSU's recommendations as described in Section 5.3.2. Nutrients will be applied to the test plots prior to seeding.

6.2.3 Seeding and Maintenance

Based on the recommendation of the Soil Science Department of Michigan State University, the recommended seed mixture will be applied to all of the test plots. After the test plots are seeded, a wheat/straw cover will be placed on the test plots to control erosion and retain moisture in the soil. At this time, a watering schedule will be established to maintain and support the growth of the grass on each test plot.

7.0 DATA MANAGEMENT

An adequate data management system is necessary to allow all personnel involved in the project easy entry and access to data for validation, interpretation, analysis and decision making purposes. The following sections describe the procedures that will be used in documenting, tracking, organizing, and presenting all data collected during the Granite City project to ensure they are of sufficient quality to provide an accurate site characterization.

7.1 Field Activity Documentation

Documentation of activities is important to provide an accurate account of work performed in the field during this study. The following methods will be used to record data:

7.1.1 Daily Logbooks

The field supervisor will be responsible for maintaining a daily logbook and sample collection documentation for field activities. Information from individual field personnel, who have been designated to perform specific tasks, will be collected and maintained by the field supervisor. Observations made by individual field personnel will be promptly recorded in the logbook. Results from laboratory or field equipment calibrations will also be recorded in the logbook. The original records will be retained for the project files.

Logbook entries will contain accurate descriptions and documentation of an individual's field activities. Logbooks will form the basis for the written reports, therefore, they will contain only facts and observations. Entries will be signed and dated by the individual making the entry on a daily basis.

7.1.2 Record Validation

Before the release of sampling documentation, project personnel will assemble all records and cross-check the information on corresponding sample labels, sample data sheets, custody records, logbooks, and other accountable documents to ensure that data pertaining to each particular sample is complete and consistent throughout the record. Examples of the sample labels, sample data sheets and NTH chain-of-custody are provided as Figures 3, 4 and 5.

If an error is made on an accountable document assigned to an individual, the individual will make corrections by crossing a line through the error and entering the correct information. Any subsequent error discovered on an accountable document will be corrected by the person who made the entry. All corrections will be initialed and dated.